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**Sintering Behavior And Mechanical Properties Of Stainless Steels Fabricated
By Powder Bed Binder Jet Printing**

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Département de génie mécanique

Mémoire présenté en vue de l'obtention du diplôme de *Philosophiæ Doctor*

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**Sintering Behavior And Mechanical Properties Of Stainless Steels Fabricated
By Powder Bed Binder Jet Printing**

présentée par **Khadijeh ESMATI**

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RÉSUMÉ

La fabrication additive par jet de liant (Binder Jet Additive Manufacturing, BJAM) est un procédé de fabrication additive fondé sur des poudres, dans lequel un liant liquide est déposé de manière sélective sur un lit de poudre afin de former une pièce verte tridimensionnelle. Comparée aux technologies de fabrication additive basées sur le laser, la BJAM offre des cadences de production nettement supérieures (jusqu'à 100 fois plus rapides), ce qui la rend particulièrement attractive pour les applications automobiles à haut débit. Après l'impression, les composants obtenus nécessitent un traitement thermique de déliantage et de frittage afin d'atteindre la densification finale. L'étape de frittage introduit cependant des défis majeurs, notamment la présence de porosités résiduelles, un retrait volumique supérieur à 30 %, ainsi qu'un affaissement induit par la gravité à haute température, ce qui entraîne des distorsions géométriques. De plus, un comportement de frittage anisotrope apparaît en raison de l'alignement préférentiel des pores généré durant le procédé BJAM, ce qui rend le contrôle des dimensions finales et des propriétés mécaniques particulièrement complexe. Une compréhension approfondie du comportement de frittage anisotrope est donc essentielle pour permettre l'intégration fiable de la technologie de binder jetting dans les applications industrielles. Par ailleurs, cette compréhension est indispensable au développement de modèles de frittage capables d'améliorer la précision dimensionnelle et les performances mécaniques des pièces imprimées. Un autre enjeu majeur dans l'industrie automobile concerne les sollicitations à haute vitesse de déformation lors des collisions et des impacts. Les pièces fabriquées par BJAM constituent des candidates prometteuses pour répondre à cette problématique. Toutefois, les études existantes se sont principalement concentrées sur l'influence des paramètres de procédé sur les propriétés mécaniques en régime quasi-statique, tandis que le comportement dynamique et la réponse aux impacts des matériaux BJAM demeurent largement inexplorés, soulignant la nécessité d'investigations complémentaires.

Les aciers inoxydables austénitiques 316L et durcissables par précipitation 17-4 PH présentent un intérêt particulier en raison de leur excellente résistance à la corrosion, de leur bon rapport résistance/masse et de leur aptitude à la fabrication, ce qui les rend adaptés aux applications automobiles et d'ingénierie avancée. Cette thèse vise à comprendre le comportement de frittage anisotrope et les défis liés à la porosité dans la fabrication additive par jet de liant, ainsi qu'à évaluer la réponse mécanique à haute vitesse de déformation des aciers inoxydables imprimés par binder jet pour des applications automobiles liées à la sécurité en cas d'impact.

Le comportement de frittage anisotrope des aciers inoxydables fabriqués par binder jet a été étudié à l'aide des alliages 316L austénitique et 17-4 PH durcissable par précipitation. La dilatométrie in situ, combinée à une caractérisation microstructurale, a permis de quantifier la cinétique de retrait en fonction de la température de frittage et de l'orientation d'impression. Les deux matériaux présentent un retrait quasi-isotrope dans le plan du lit de poudre, tandis qu'une anisotropie marquée se développe selon la direction de construction, en raison de l'architecture des pores induite par l'impression. Des lignes primitives orientées perpendiculairement à la direction de construction agissent comme des chemins préférentiels d'effondrement pendant le frittage. Avec l'augmentation de la température, une transition d'un mécanisme de diffusion aux joints de grains à une diffusion volumique accélère la densification dans les deux alliages. Dans le 316L, la formation de δ -ferrite au-delà d'environ 1300 °C augmente la diffusivité et accroît le retrait dans la direction de construction jusqu'à environ 15–16 %. Dans le 17-4 PH, l'évolution de phase et la densification contrôlée par diffusion volumique au-delà d'environ 1250 °C conduisent à une anisotropie plus prononcée, avec un retrait dans la direction de construction pouvant atteindre près de 40 %. L'énergie d'activation de la densification, la taille de grain et la densité finale montrent une dépendance limitée à l'orientation d'impression, indiquant que le retrait anisotrope est principalement gouverné par l'effondrement des lignes primitives, l'évolution des pores dépendante de la température et la formation de phases, plutôt que par des cinétiques de diffusion dépendantes de l'orientation. Un modèle viscoplastique de frittage est également utilisé pour interpréter et prédire le comportement de frittage anisotrope d'une pièce en 17-4 PH fabriquée par binder jet, en reliant la distribution des pores, la température et l'évolution des propriétés du matériau au retrait observé. Basé sur un cadre de densification contrôlée par diffusion avec une viscosité dépendante de la direction, le modèle reproduit avec succès les réponses distinctes de retrait selon la direction de construction et le plan in-plane.

La seconde partie de cette thèse examine l'effet de la distribution granulométrique des poudres (PSD) sur le comportement de frittage anisotrope du 316L fabriqué par binder jet. Des poudres issues de la découpe MIM (PSD étroite) et de la découpe LPBF (PSD plus large) ont été traitées dans des conditions de frittage identiques afin d'isoler le rôle de la PSD sur la densification et le retrait. Les résultats montrent que les poudres MIM présentent un retrait linéaire plus important dans la direction de construction et atteignent une densité finale plus élevée à des températures de frittage plus faibles en raison de leur taille de particule plus fine et de leur PSD plus étroite. De

manière cohérente, les échantillons MIM développent une taille de grain plus fine que les échantillons LPBF, la PSD plus fine favorisant une densification plus rapide, renforçant l'ancrage des joints de grains et limitant l'exposition thermique. Le retrait total, plus élevé avec les poudres MIM, entraîne une anisotropie de frittage plus prononcée. En comparaison, les échantillons LPBF bénéficient de leur PSD plus large et d'une meilleure densité d'empilement des poudres, ce qui se traduit par une anisotropie de retrait plus faible et une meilleure stabilité dimensionnelle durant le frittage.

La dernière partie de cette thèse étudie le comportement en compression à haute vitesse de déformation du 316L, fabriqué par binder jet, dans des conditions de sollicitation représentatives des collisions, à l'aide d'essais de type Split Hopkinson Pressure Bar. Les effets de la vitesse de déformation, de la température, de la distribution granulométrique des poudres et du traitement par compression isostatique à chaud (HIP) sont évalués de manière systématique. Les résultats montrent que l'augmentation de la vitesse de déformation accroît la contrainte d'écoulement, tandis que l'élévation de la température induit un adoucissement thermique significatif. La distribution granulométrique et le traitement HIP influencent la densité et la taille de grain, mais ont un effet limité sur la résistance à la déformation à haute vitesse. Pour décrire le comportement thermovisco-plastique observé, des modèles constitutifs phénoménologiques, notamment les formulations de Johnson–Cook et de Hensel–Spittel, ainsi que leurs versions modifiées, ont été calibrés et évalués. Les modèles modifiés améliorent la prédiction du comportement contrainte–déformation sur une large gamme de vitesses de déformation et de températures.

ABSTRACT

Binder jet additive manufacturing (BJAM) is a powder-based AM process in which a liquid binder is selectively deposited onto a powder bed to form a three-dimensional green part. Compared with laser-based AM technologies, BJAM offers substantially higher production rates (100x faster), making it particularly attractive for high-throughput automotive applications. Following printing, BJAM components require a debinding and sintering heat treatment to achieve densification. The sintering step introduces significant challenges, including residual pores, volumetric shrinkage exceeding 30%, and gravity-induced sagging at elevated temperatures, which lead to geometric distortion. Additionally, anisotropic sintering behavior arises from the preferential alignment of pores during the BJAM process, making it challenging to control the final dimensions and mechanical properties of the parts. Therefore, a comprehensive understanding of anisotropic sintering behavior is required to enable the reliable integration of binder jetting technology into industrial applications. Moreover, such knowledge is essential for developing a sintering model that improves the dimensional accuracy and mechanical performance of printed parts. One of the key challenges in the automotive industry is high-strain-rate loading during crashes and impacts. Parts manufactured using BJAM are promising candidates for addressing this challenge. However, existing studies have primarily focused on the influence of BJAM processing parameters on quasi-static mechanical properties, while the impact behavior and dynamic response of BJAM materials remain largely underexplored, highlighting the need for further investigation.

Austenitic 316L and precipitation-hardened 17-4 PH stainless steels are of particular interest due to their excellent corrosion resistance, favorable strength-to-weight ratio, and good manufacturability, making them ideal for advanced automotive and engineering applications. This thesis focuses on understanding anisotropic sintering behavior and porosity-related challenges in binder-jet additive manufacturing, and on evaluating the high-strain-rate mechanical response of binder-jetted stainless steels for crashworthiness-relevant automotive applications.

The anisotropic sintering behavior of binder-jetted stainless steels was investigated using austenitic 316L and precipitation-hardened 17-4 PH alloys. In situ dilatometry, combined with microstructural characterization, was employed to quantify shrinkage kinetics as a function of sintering temperature and printing orientation. Both materials exhibit quasi-isotropic shrinkage within the powder bed plane, whereas pronounced anisotropy develops along the build direction

due to printing-induced pore architecture. Primitive lines aligned normal to the build direction act as preferential collapse paths during sintering. With increasing temperature, a transition from grain-boundary to volume diffusion accelerates densification in both alloys. In 316L, δ -ferrite formation above ~ 1300 °C enhances diffusivity and increases build-direction shrinkage to ~ 15 – 16 %. In 17-4 PH material, phase evolution and volume-diffusion-controlled densification above ~ 1250 °C result in stronger anisotropy, with build-direction shrinkage approaching 40 %.

The activation energy for densification, grain size, and final density show limited dependence on printing orientation, indicating that anisotropic shrinkage is governed by primitive-line collapse, temperature-dependent pore evolution, and phase formation rather than orientation-dependent diffusion kinetics. A viscoplastic sintering model is also employed to interpret and predict anisotropic sintering in a 17-4 PH binder-jetted part by relating pore distribution, temperature, and material-property evolution to shrinkage. Built on a diffusion-controlled densification framework with direction-dependent viscosity, the model successfully captures the distinct shrinkage responses along the build and in-plane directions.

The second part of this thesis investigates the effect of powder size distribution (PSD) on anisotropic sintering behavior in binder-jetted 316L stainless steel. Metal injection molding (MIM)-cut powders with a narrow PSD and laser powder bed diffusion (LPBF)-cut powders with a broader PSD were processed under identical sintering conditions to isolate the role of PSD on densification and shrinkage. The results show that MIM-cut powders exhibit greater linear shrinkage along the build direction and achieve higher final density at lower sintering temperatures due to their finer particle size and narrower PSD. Consistent with this behavior, MIM-cut samples develop a finer grain size than LPBF-cut samples, as the finer PSD promotes faster densification, enhances grain boundary pinning, and limits thermal exposure. The greater total sintering shrinkage associated with MIM-cut powders results in more pronounced anisotropic sintering. In comparison, LPBF-cut samples benefit from their broader powder size distribution and more powder packing density, resulting in lower shrinkage anisotropy and improved dimensional stability during sintering.

The final part of this thesis investigates the high-strain-rate compressive behavior of binder-jetted 316L stainless steel under crash-relevant loading conditions using Split Hopkinson Pressure Bar testing. The effects of strain rate, temperature, powder size distribution, and hot isostatic pressing

(HIP) are evaluated. Results demonstrate that strain rate increases flow stress, while temperature induces significant thermal softening. Powder size distribution and HIP treatment influence density and grain size but have a limited effect on high-strain-rate strength. To describe the observed thermo-viscoplastic behavior, phenomenological constitutive models, including Johnson–Cook and Hensel–Spittel formulations and their modified versions, are calibrated and assessed. The modified models improve the prediction of stress–strain behavior over a wide range of strain rates and temperatures.

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CHAPTER 1 INTRODUCTION

Binder jetting (BJ) is an additive manufacturing (AM) process used to construct three-dimensional (3D) metal structures with complex geometries [1]. Parts are constructed by depositing a liquid binding agent layer by layer using computer-aided design (CAD) [2]. Subsequently, the printed parts undergo a three-step post-heat treatment to produce fully dense, near-net-shape components [3,4]. These post-processing steps, including curing, debinding, and sintering, are necessary to achieve the desired mechanical properties. In the first step, the printed parts are cured in a convection oven to remove excess liquid binder and to strengthen the bonding between the binder and powder particles through cross-linking and further polymerization [5]. The debinding step involves controlled heating, with the debinding temperature chosen based on the binder's chemical composition and thermal decomposition behavior [6]. An appropriate heating cycle is required to ensure complete binder removal and to prevent residual species, such as carbon. The final step is sintering, a thermal process in which powder particles are bonded into a solid structure through atomic-scale diffusion without complete melting [7]. Key parameters include sintering temperature and time, heating rate, atmosphere, and powder characteristics such as chemical composition, particle size, and shape.

Binder jetting (BJ) is a high-throughput additive manufacturing process that enables rapid layer deposition over large build areas and the simultaneous production of multiple parts [8,9]. Moreover, BJ allows the fabrication of geometrically complex components, such as parts with internal channels and integrated functionalities, which are challenging to produce using conventional manufacturing techniques. These characteristics make BJ well-suited for the automotive industry, where manufacturing is driven by low margins and the need for high-volume production [8,9].

As illustrated in Fig. 1, BJ can be applied to both large, geometrically complex structural components, such as an engine block (Fig. 1a) [9], and functional components with performance-critical features like a piston (Fig. 1b) [10], demonstrating the scalability and versatility of the process for automotive applications.

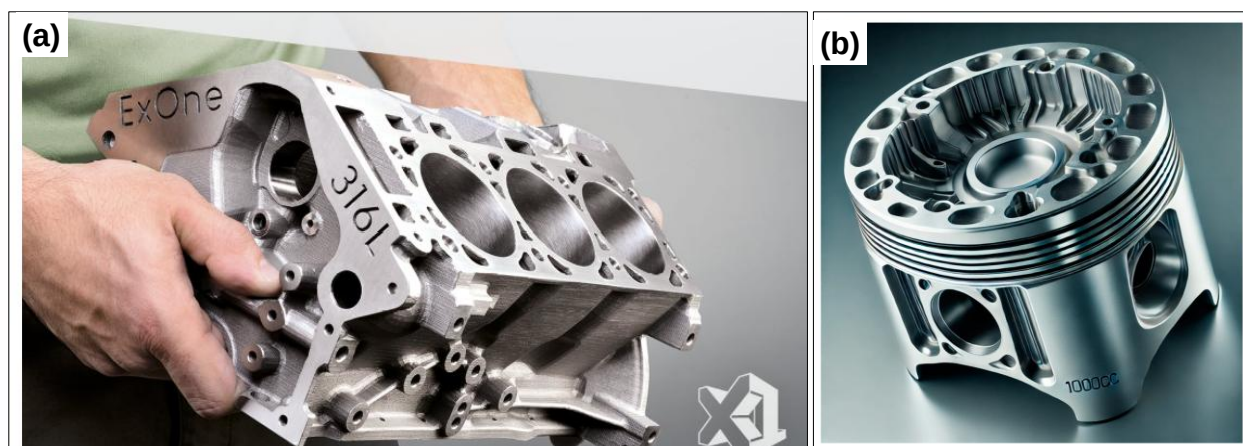


Fig.1-1. BJ additively manufactured automotive components (a) engine block [9] and (b) piston [10].

The BJ process is a cost-effective and time-saving method for producing automotive manufacturing parts and structural components [8,9,11]. However, during sintering, binder-jetted parts undergo significant shrinkage ($\sim 20\%$) to achieve a high relative density [12]. The sintering shrinkage is often anisotropic due to layer-by-layer deposition, heterogeneous pore distribution, and directional microstructural features introduced during printing [13]. Therefore, accurate prediction of total shrinkage is essential for achieving final parts dimensions. Existing shrinkage models assume isotropic behavior, and they fail to capture the anisotropic nature of BJAM sintering [14,15]. As a result, an anisotropic shrinkage model is required to more reliably predict the final dimensions of printed parts. In addition, the influence of the binder jet and sintering parameters, including material composition, printing orientation, powder size distribution, sintering temperature, and phase evolution on anisotropic sintering shrinkage, remains poorly understood.

Parts manufactured by the binder jetting (BJ) method must demonstrate reliable mechanical performance under service-relevant loading conditions [16]. This requirement is especially critical for automotive and structural components, which commonly experience high-strain-rate loading during impact events. However, the influence of printing and sintering parameters on the dynamic mechanical behavior of binder-jetted parts remains insufficiently explored.

Stainless steel is well-suited for automotive and structural applications due to its high corrosion resistance, thermal stability, and mechanical reliability [17]. Austenitic 316L and precipitation-hardened 17-4 PH are two particularly promising candidates for industrial binder jetting applications. Austenitic 316L offers excellent corrosion resistance, high ductility, and superior

energy absorption, making it suitable for safety-critical components that requireable deformation under impact loading [18–20]. In contrast, precipitation-hardened 17-4 PH offers high strength, good fatigue resistance, and thermal stability after heat treatment, enabling lightweight, load-bearing components where dimensional accuracy and mechanical efficiency are critical [21,22].

This thesis is organized as follows: a comprehensive literature review is presented in Chapters 2 and 3, establishing the scientific background of this work. The scientific approach adopted in this thesis is described in Chapter 4. Chapters 5 to 8 present the main scientific contributions of this work in the form of three research articles. The findings of this study are summarized in Chapter 9, where the study's limitations and directions for future research are also discussed.

CHAPTER 2 LITERATURE REVIEW ON BINDER JETTING

2.1 General process description

Binder jetting (BJ) is an additive manufacturing method that deposits a liquid binder into predetermined areas of a digital 3D model. As shown in Figure 2-1, CAD software was first used to create a 3D drawing of the original specimen, which was then imported into the BJ machine. After dispersing a layer of powder, a printhead selectively distributes a predetermined number of binder droplets over the powder bed's surface. The process is repeated until the complete component (also known as the green parts) is printed. The green parts are heated to cure the binder phase and produce minimum mechanical strength. The green component is then heated in the furnace to further break down the residual binder phases. Due to the low mechanical properties of green parts, they will undergo additional post-processing, such as infiltration and sintering, to provide sufficient mechanical strength [23].

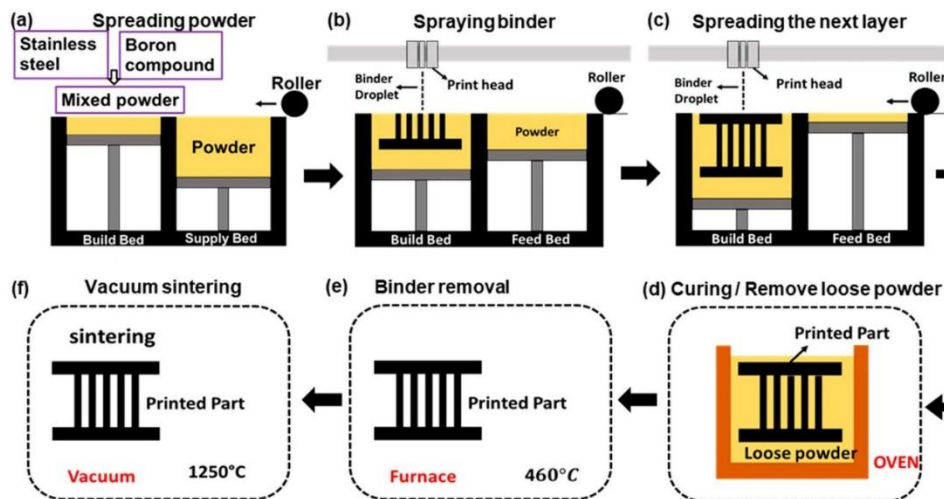


Figure 2-1 Binder jetting printing procedure (BJ) [23].

2.1.1 Benefits and challenges of the binder jetting process

In comparison to other additive manufacturing technologies, the following are the key benefits of the binder jetting process:

1. Contrary to powder bed fusion (PBF) procedures, in which metal powders are melted by laser or electron beam, the BJ process uses no power heat sources, allowing it to work with various materials [24].

2. Support structures are not required in the binder jet process because the components in the powder bed are self-supporting. Stability in fusion-based additive manufacturing (AM) requires the creation of support material and its attachment to the build plate.
3. In the binder-Jetting technique, parts can be printed at room temperature. Therefore, this ability leads to greater design flexibility and less residual stress.
4. Binder jetting printers may create parts up to 100 times quicker than laser bed fusion systems as they use a single print head to jet the binding agent onto the powder bed in a single pass, rather than building up the part layer by layer [25].
5. Unlike other approaches, binder jetting also makes it possible for batch production, which lets companies print numerous variations of a single part at once, thus lowering part costs. In contrast, the selection of materials for laser-based devices is constrained. They can spend up to \$60 per kilogram on materials because they can only work with low-oxygen metal powders. The completed parts can cost hundreds of dollars per kilogram, making them much too expensive for large-scale manufacturing. The production system may recycle as much as 99% of wasted powder thanks to binder jet systems, which reduce waste and increase cost-effectiveness [26].

The two main drawbacks of binder jetting can be mentioned as follows:

1. The relative density of as-printed parts is lower (50%) than that of fusion-based methods, and densification from this condition typically causes considerable geometry deformation and shrinkage [27].
2. In BJ, post-processing is one of the most important and necessary steps to achieve the required density and mechanical properties.

2.1.2 Current status and application of the binder jetting

Different industries will become more interested in binder jetting as technological advances allow for the highly accurate sintering of nanoscale structures, cantilevered structures, trusses, and microchannel heat. Binder jetting has recently been used for projects like producing biopolymers [28], electronic devices [29], battery storage [30], and electrochemical energy storage [31]. Although in the beginning, less attention and investment were paid to binder jetting than LPBF processes, some companies like ExOne have collaborated with numerous organizations and businesses on binder jetting research since the 1990s to provide process parameters for printing

and densifying famous ceramics and metals [32], [33]. These days, BJ has been applied to a range of materials, including stainless steel [34], copper [35], aluminum [36], titanium [37], and nickel-based superalloys [38]. In the future, a potentially impactful application of BJ for battery electric vehicles (BEVs) will be the production of novel multi-functional battery casings which support the battery cells, protect them from impact, and provide thermal management solutions [39].

2.1.3 Machine description

Figure 2-2 shows the schematic of a BJ printer, including a hopper, roller, printhead, and heat lamp. The process begins by dispersing powder from the hopper onto the build platform. Then, the roller moves across the platform to form a uniform layer of powder. Next, the binder is deposited onto the powder bed using the printhead guided by the 3D model. The binder adheres the powder particles together, forming a solid layer. The heat lamp passes over the build platform to dry the layer after the binder is deposited, and the job box is moved down by one layer thickness. The process is repeated until the desired number of layers is completed and the part is fully formed [40].

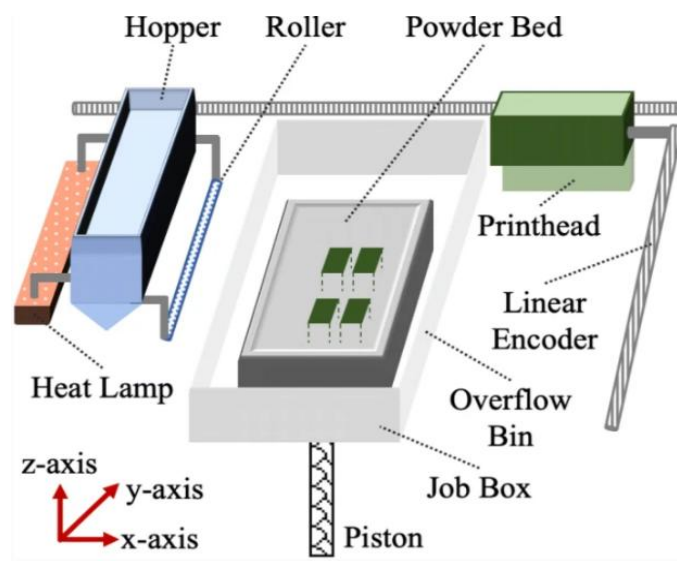


Figure 2-2 The ExOne Innovent BJ printer. Red arrows denote the printing directions [40].

2.1.4 Powders

Additive Manufacturing (AM) is a new manufacturing method divided into numerous fields. Therefore, it is essential to know the feedstocks' requirements for each Additive Manufacturing (AM) technique. It is generally accepted that understanding powder characteristics like morphology, means size, size distribution, packing density, and flow for each AM technology will

significantly affect the final parts' structure and mechanical properties [41]. Table 2.1 [42] lists common pre-alloyed powders accessible for binder jetting machines.

Table 2.1 Metallic powders commonly used in BJ [42].

Base	Common alloy name	Alloy designation (ASME)
Fe	Maraging steel	18 MAR 300
Fe	Stainless steels	316, 316L, 304, 410, 420
Fe	Hardenable tool steel	15-5
Fe	Hardenable stainless steel	17-4PH, 13-8
Fe	Tool steels	H13, S7
Ni	Superalloys	IN-718, IN-625, Waspalloy, Rene 41
Co	Superalloys	Co-Cr-Mo (ASTM F75), Stellite 6 & 21
Ti	Ti-rich	Ti-6Al-4V, Ti-6-2-4-2, Ti-6Al-7Nb
Ti	Commercial purity	CP
Al	Cast alloys	Al4047, Al-Si-10Mg, Al-12Si, Al-Si-7Mg-Cu
Cu	Bronze	Cu-Sn

The powder morphology plays a significant role in determining the efficiency of the process and the quality of the final BJ products [43]. Atomization is known as one of the important methods of powder production. The powder is often atomized using two different processes: gas atomization (GA), which results in powder with a spherical shape, and water atomization (Figure 2-3 a) (WA), which results in powder with an irregular shape (Figure 2-3 b) [44].

Spherical morphology is preferred in BJ for numerous reasons [45]. First, spherical particles also mix well with the binder material and disperse uniformly, resulting in good homogeneity in the powder bed. Second, the large surface area of a round particle increases the friction between particles in all directions, increasing the green strength of the parts. Moreover, spherical shapes can achieve high packing density and flowability in the powder bed [44].

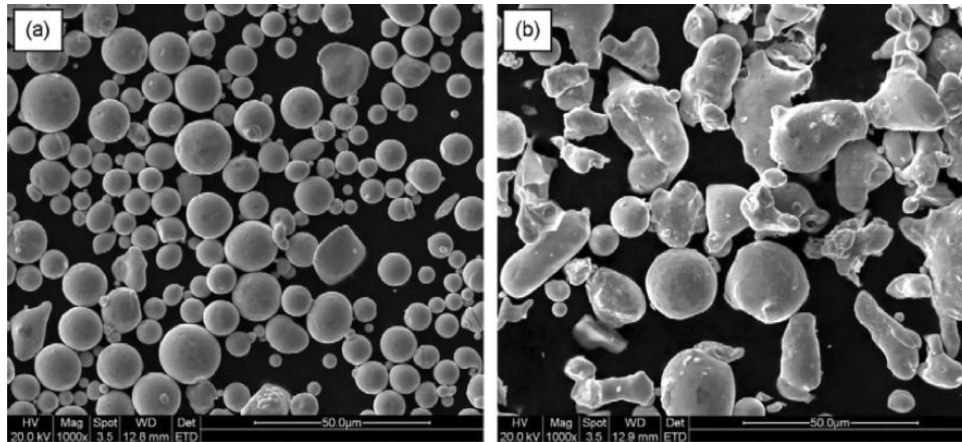


Figure 2-3 Gas atomized (a) vs. Water atomized powder SS316L (b) [44].

Coarse powder particles are preferred for BJ printing due to their improved flowability and reduced surface area; however, they have low sinterability [25]. Finer powders exhibit excellent sinterability but have poor flowability due to strong inter-particle interactions, agglomeration, and humidity. This prevents even and smooth layer deposition. Medium-sized powders attain the highest density because of the balance between flowability and sinterability [46]. Powders in BJ typically have particle sizes between 15 and 50 μm [47].

2.1.5 Binder

The binder is a crucial part of BJ because it fills the gaps between the powder layers, producing the desired shape. It must fulfill various criteria such as the appropriate rheology and stability, sufficient powder bed wetting, binding strength, and suitable burn-off, leaving the least harmful chemical traces interfering with the metallurgy [48]. Three binders can generally be arranged: (1) acid-based binders, where an acid-based reaction controls the bonding between powder particles; (2) metal salt binders, where the connection between particles is created by a reaction that involves salt recrystallization or displacement; and (3) aqueous binders, which are water or solvent-based and specifically made for polymeric and metal powders, resulting in designed structures upon solvent evaporation [49].

2.1.6 Rheology for ink jetting

Rheology is defined as a material's flow behavior under applied deformation force. Both the Weber number, We (Eq.1) [50], and the Reynolds number, Re (Eq. 2) [50], compute surface tension and viscosity. The parameters are crucial in describing the liquid binder behavior during inkjet

printing's drop creation [50]. These equations define d as the diameter of the droplet, η the liquid viscosity, σ the surface tension, ρ the liquid density, and V as the flow rate. The Ohnesorge number (Oh) (Eq.3) [50], which is the combination of (Eq.1) and (Eq.2), was used to forecast the binder wettability to ensure the binders are appropriate for inkjet printing [50]. For BJ, liquid binders with Oh values ranging from 0.1 to 1 can be employed [51,52]. When $1/Oh$ is less than 1, viscous forces are dominant inhibiting droplet separation from the liquid. A continuous column is ejected when $1/Oh$ is greater than 10, which may create satellite drops after the primary drop (Figure 2-4).

$$We = \frac{\rho d V^2}{\eta} \quad (1)$$

$$Re = \frac{\rho d V}{\sigma} \quad (2)$$

$$Oh = \frac{\sqrt{We}}{Re} = \frac{\sqrt{\eta}}{\sigma \rho d} \quad (3)$$

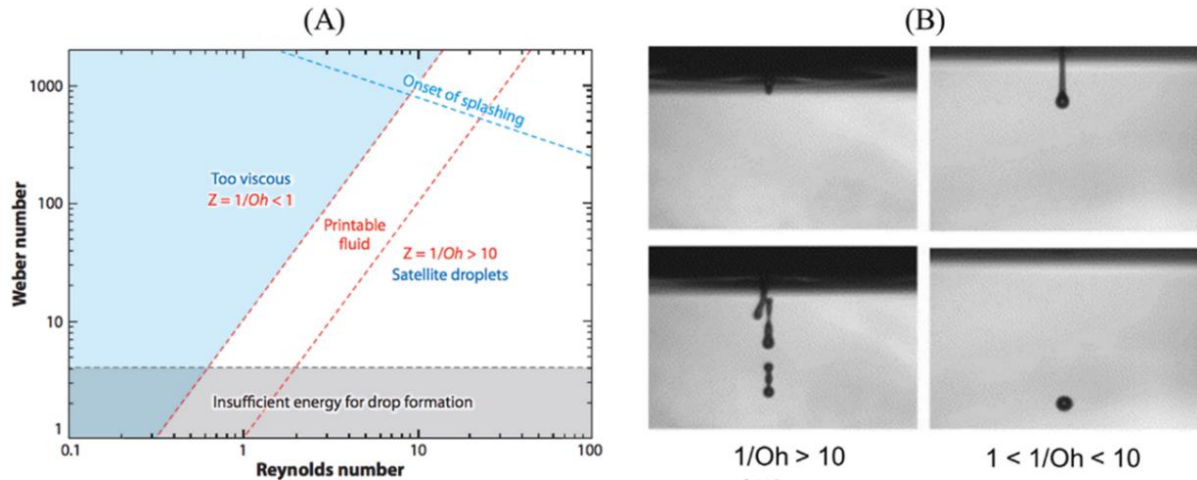


Figure 2-4 (A) A jettable zone as a function of We and Re numbers (B) Images of fluid being ejected that demonstrate the impact of the $(1/Oh)$ ratio [52].

2.2 Binder jetting printing parameters

2.2.1 Layer thickness

Layer thickness is the thickness of one powder layer deposited during recoating along the build direction. The range of layer thicknesses for BJ metal powders is between 15 and 300 μm [25]. Bai

et al. [53] suggested that the layer thickness should be up to three times the largest powder size to ensure good flowability and spreadability. As the layer thickness is reduced, the printed parts will be more delicate, and a higher final resolution will be achieved; however, the time of printing will be increased. Moreover, Zhang et al. [54] showed that a thicker layer in BJ resulted in more porosity, a rougher surface finish, and decreased mechanical properties.

2.2.2 Binder saturation

The term "printing saturation" or "binder saturation" refers to the percentage of air volume the binder fills, as defined in Eq. 4 [55]. The printing saturation must be at its optimum level [56]. In low saturation (Figure 2-5 a), the samples may collapse because of insufficient binder to connect the powders firmly. In contrast, a high saturation value may affect the part's dimension due to powder joining (Figure 2-5 b). The printing saturation level affects the surface of the printed item [57]. The surface roughness will be poor if the printing saturation level is higher or lower than the default value. In the lower level, an insufficient binder creates a solid binding between powder particles. In this case, a small amount of powder ejecting from the printhead results in a zigzag pattern and undesirable surface roughness. In the higher-than-optimal situation, excessive powder bonds to the surface, resulting in surface swelling and poor surface roughness [58].

$$S = \frac{V_{binder}}{V_{air}} = \frac{V_{binder}}{\left(1 - \frac{V_{powder}}{V_{powder} + V_{air}}\right) \times V_{solid}} \quad (4)$$

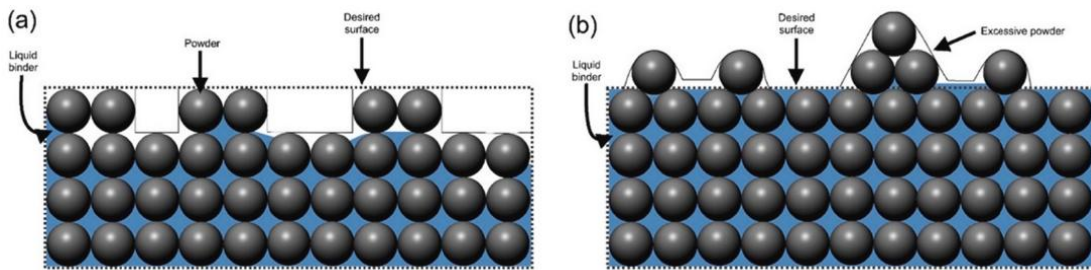


Figure 2-5 Printing saturation: a) low saturation and b) high saturation [56].

2.2.3 Drying time

Drying time is the time required for the binder to dry under a heater after deposition onto the powder bed. Short drying time causes printhead clogging, significantly affecting the finished product's surface quality [55]. Printhead clogging occurs when the printhead does not have enough time to remove the excess binder.

Figure 2-6 shows the effect of drying time on the final quality of printed parts [59]. Generally speaking, when the binder has enough time to dry (Figure 2-6 (a)), previously dried layers restrict the new binder's vertical penetration (along the build direction (BD)). In this case, binder penetration primarily occurs in the new layer between the deposited droplets in unsaturated areas, which improves the part's dimensional accuracy and green strength. Figure 2-6 (b) shows a part printed with a short drying time. The binder exhibits higher penetration in the vertical direction than in the thickness direction. This leaves some of the binders uncured, causing dimensional inaccuracies and weakening the structure [59].

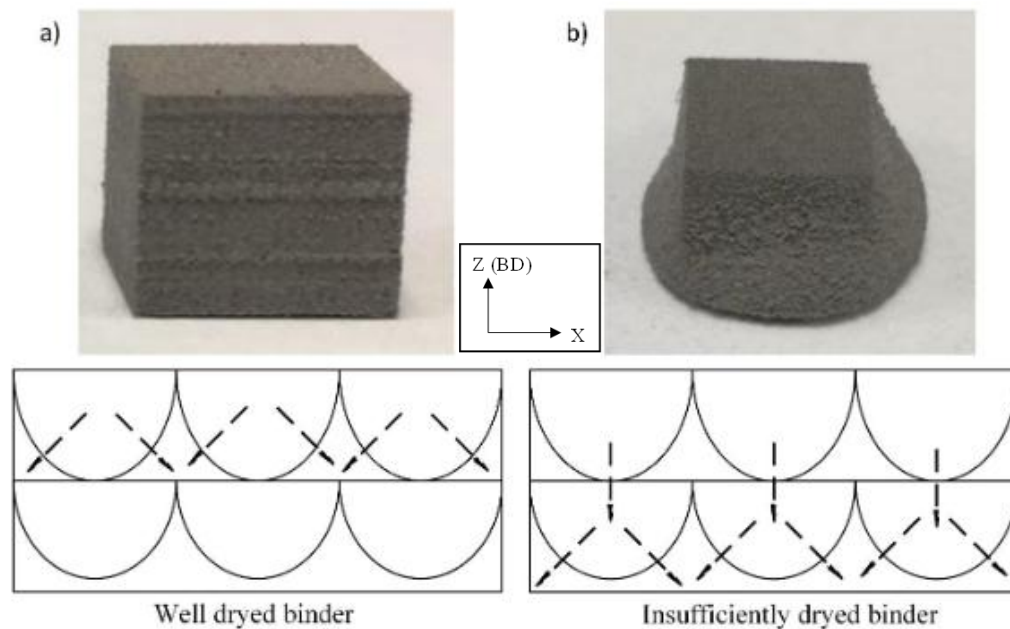


Figure 2-6 Effect of drying time on the quality of Ti-6Al-4V BJ printed parts: a) proper drying time and b) inadequate drying time (Dash lines depict the potential migratory routes of the binder within the powder bed.) [59].

2.3 Binder jetting post-processing chain

Post-processing steps such as binder curing, de-powdering, de-binding, and sintering are necessary to acquire the desired mechanical characteristics. Infiltration or hot isostatic pressing (HIP) can be applied as an extra post-processing step to enhance the part's structural integrity and density.

2.3.1 Curing and De-powdering

Printed parts are cured together to eliminate extra liquid binders and strengthen the bond between the binder and powders by cross-linking and further polymerization (Figure 2-7) [5]. The curing temperature and time are important factors that depend on the binder type and material used. In BJ, drying takes place during printing after the binder is sprayed. In contrast, curing takes place at the end of the process at a higher temperature and for a longer time to remove all excess binders and increase the mechanical strength of the printed parts. Several techniques to complete the curing process include heating the component in an oven, applying UV radiation, or combining both [5].

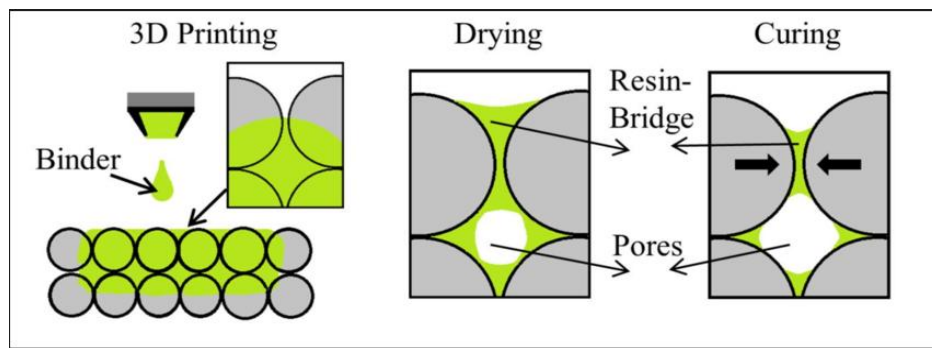


Figure 2-7 Schematic of the curing process in BJ [5].

Depowdering is a process used in binder jetting to remove the excess powder left on the surface of the printed part after the curing [55]. This step is typically done by blowing compressed air and using a brush or vibrating the table to remove the powder. Depowdering is important because excess powder can generate porosity or reduce the strength of the final part. This process ensures that the printed part is ready for post-processing or final use [25].

2.3.2 Debinding

Debinding occurs during the first sintering stage when the binder becomes weak and breaks down into evaporable components. Debinding is necessary because, without it, any remaining carbon and other harmful chemical substances from the binder could diffuse inside the part during sintering

and deteriorate its mechanical properties [24]. The key parameters to govern the process are the de-binding temperature, hold time, and gas flow. The thermal debinding temperature varies depending on the metal and binder employed but typically occurs between 200 °C and 650 °C [25,52]. The easiest way to determine the hold temperatures for the binders is to perform a thermo-gravimetric analysis (TGA) of the feedstock. Typically, the heating rate for the TGA process is similar to the thermal debinding process in the furnace. An example curve from a TGA experiment using a 4140 wax-based feedstock is shown in Figure 2-8[60]. Based on this analysis, the holding temperatures were found to be 380, 440, and 540 °C. Isothermal holding times are used at these temperatures to eliminate all binders from the component. The holding time is chosen based on the part size, binder composition, and binder quantity [45]. Gas is continuously purged into the furnace to remove the binder and ensure the air does not oxidize the metal. If there is still a binder at the end of the de-binding process, de-binding may be incomplete. As the component is heated to a higher temperature for sintering, the residual binder quickly evaporates. Defective parts and soot buildup on the furnace's heating element or inside the sample can stem from insufficient de-binding [60].

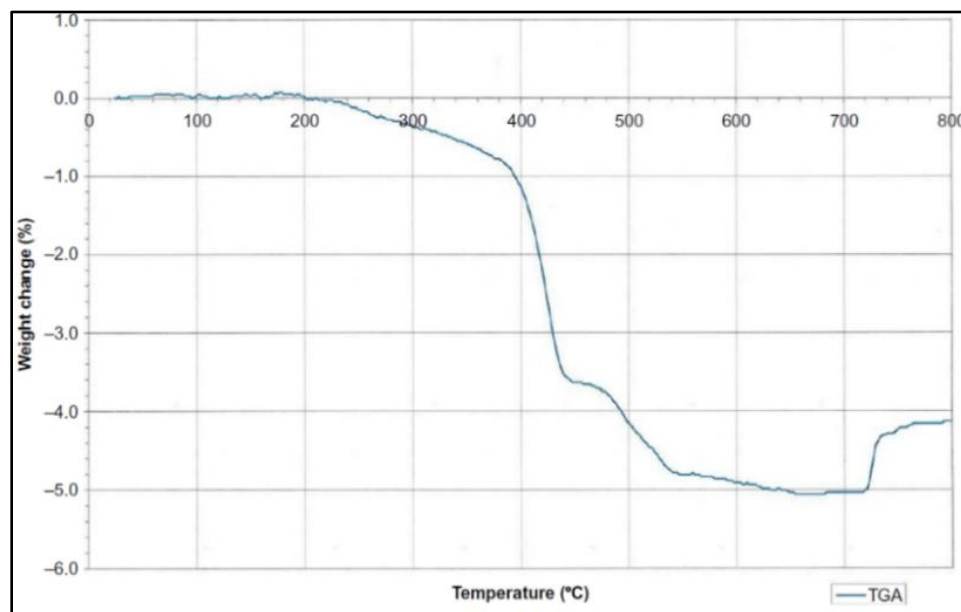


Figure 2-8 TGA results for 4140 MIM (Metal Injection Molding) feedstock. Three different de-binding temperatures include (1) 380°C, (2) 440°C, and (3) 540°C (Heaney, 2019).

2.3.3 Sintering

Sintering is a thermal process that uses atomic-scale mass transport or diffusion to bond particles into solid structures [6]. High surface energy and the curved surface inherent to a powder have acted as the driving forces for sintering. In general, producing the sintering component with appropriate qualities requires controlling the density, grain size, and porosity distribution. There are two important factors in the sintering process: process factors and material [61]. The process factors include temperature and time of sintering, heating rates, sintering atmospheres, and the material variables, such as chemical composition, size, shape, and other powder characteristics [7].

The three main stages in solid-state sintering are the initial, intermediate, and final stages (Figure 2-9). The initial stage is characterized by irregular pores and the creation of necks between powders. Densification in this stage only accounts for 2-3% of the total densification [7]. The intermediate stage includes open and closed pores. Mass continues to be transferred along pore curves to fill up open areas. Necks might interfere with one another and overlap at this stage. Particle rearrangement is the principal mechanism by which grains expand and pores close. Before the isolation of the pores, the relative density can reach up to 93% [55]. In the final stage, most pores are closed and distributed mostly inside the grain. After this stage, grain growth accelerates as there are insufficient pores in the grain boundary to inhibit this phenomenon [7].

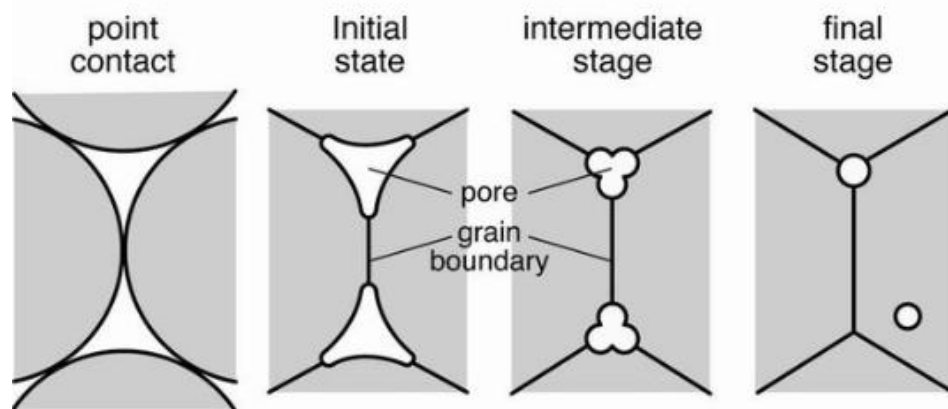


Figure 2-9 Three main stages in solid-state sintering [7].

2.3.4 Post-sintering: Infiltration and HIP

Infiltration is one of the most commonly used methods for post-sintering to achieve nearly full density [62]. This procedure might be applied if the composition does not impair the component performance and the part's mechanical properties. In the infiltration process, the infiltrant's melting temperature must be lower than the base material's melting point or solidus temperature to avoid the part's structural integrity loss [62]. In BJ SS 316L, bronze is mostly utilized as an infiltrant because of its low melting temperature [55]. In addition, infiltration can enhance the mechanical properties of a sintered BJ part because it fills the interparticle gaps and removes the potential area for stress concentration [55]. The HIP process is another popular post-sintering method used to achieve full density [63]. The printed parts are subjected to high temperature and pressure in a controlled environment to remove internal porosity and create a more uniform, dense material structure. Unlike infiltration, it is used for sections with isolated porosity with over 92 % relative density and at temperatures lower than those used in sintering [25,55].

2.4 Sintering model and densification

2.4.1 Thermodynamics of sintering

The thermodynamic driving forces for sintering include surface energy, interfacial energy, and curvature gradients [64]. Surface energy is especially important to join the particles during sintering. Smaller particles sinter more quickly than larger ones due to higher surface energies [7]. High temperatures also accelerate sintering because atomic mobility increases with temperature. Figure 2-10 shows a two-sphere sintering model developed by Frenkel [65]. The surface area reduces during the sintering process until the system reaches the minimal total surface energy required to achieve thermodynamic equilibrium.

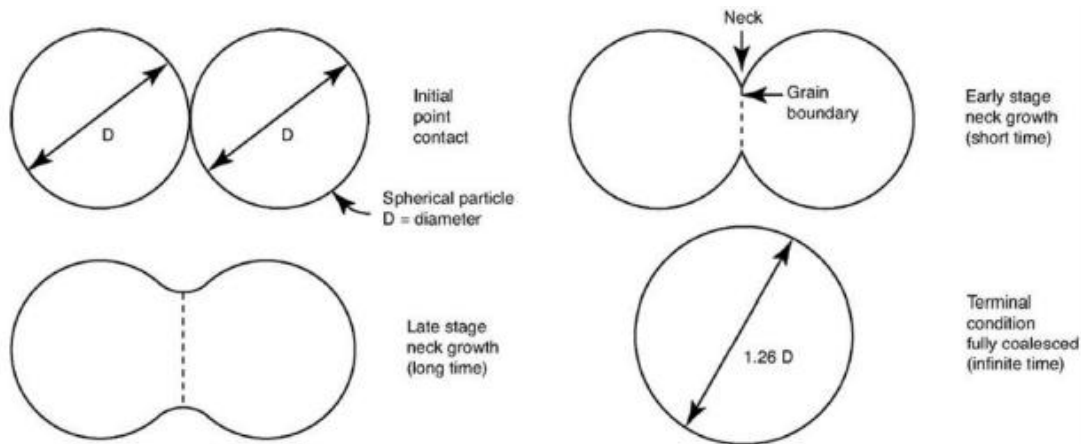


Figure 2-10 The surface area reduction during sintering based on the two-sphere sintering model [7].

2.4.2 Solid-state sintering

In solid-state sintering, particles stay solid and do not melt [64]. Typically, the green samples are heated up to a temperature of 0.5–0.9 of the material's melting point and kept there for some time, depending on the material's size and ease of densification [66]. The appropriate microstructure can be achieved through mass transport via solid-state diffusion [64]. In crystalline solids, transport occurs by diffusion of atoms, ions, or molecules along concrete paths that define the mechanisms of sintering. According to Figure 2-11, at least six different processes or tools can lead to the sintering of crystalline materials: vapor transport, surface diffusion, lattice diffusion (from the surface), grain boundary diffusion, plastic flow, and lattice diffusion (from the grain boundary) [64]. The sintering mechanisms are divided into two categories: non-densifying mechanisms and densifying mechanisms seen in Figure 2-11. Shrinkage and compaction are caused by some material transfer mechanisms, such as grain boundary diffusion, network (volumetric) diffusion, and plastic flow. Other mechanisms, such as vapor transport, surface diffusion, and volume diffusion, cause neck growth and particle coarsening without compaction. The result will be a porous microstructure if non-dense mechanisms predominate [64,66].

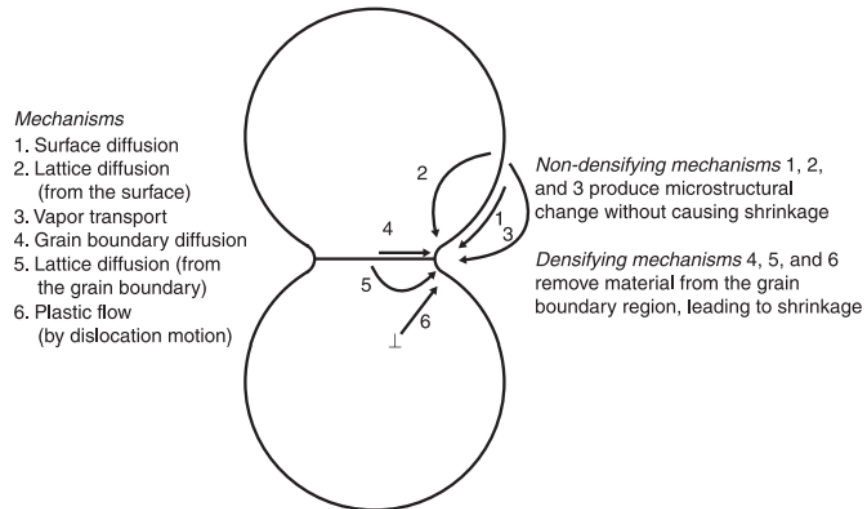


Figure 2-11 Material transport pass during sintering [64].

2.4.3 Stages of sintering

The sintering process is typically classified into three stages: initial, intermediate, and final stages, each characterized by distinct densification and microstructural evolution mechanisms, as discussed further.

In the initial sintering stage, necks start forming at the contact area between adjacent particles. This stage is known as the "neck growth" stage, where the neck size ratio (X/a) must be less than 0.3 [66]. The geometrical model for two particles is shown in Figure 2-12 where (X) is the radius of the neck, and (a) is the particle's radius. Figure 2-12 (a) shows the initial sintering stage without shrinkage. In this stage, the neck size grows with increasing sintering time, but the distance between particles remains constant. During sintering, surface diffusion causes a decrease in the surface area without shrinkage. However, Figure 2-12 (b) exhibits shrinkage because bulk transport (volume diffusion and grain boundary diffusion) between the particles causes neck growth as sintering time increases [64,66].

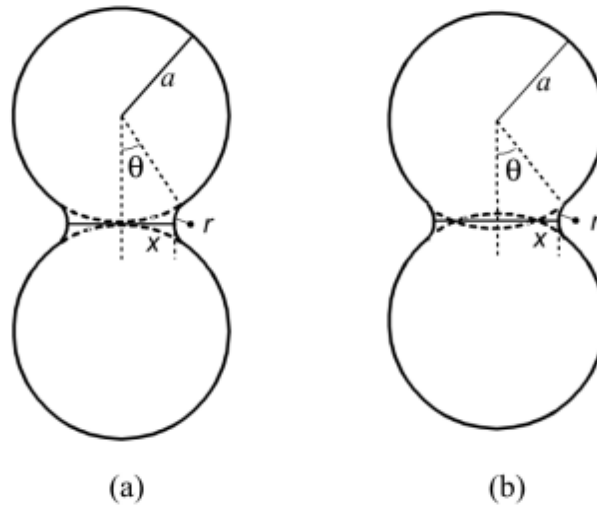


Figure 2-12 Two-particle sintering models: (a) without shrinkage and (b) with shrinkage [66].

The intermediate stage is crucial for densification and the final characteristics. The driving forces for sintering include removing interfacial, surface, and grain boundary areas. A mixture of densification, pore rounding, and grain expansion occurs throughout this stage [67]. The fractional density (f) is used to relate the pore diameter (d) and grain size G for any shape, as shown in (Eq. 5) [61]. As long as the pores are in contact with the grain boundaries, Eq. 5 is valid.

$$f = 1 - 2\pi(d/G)^2 \quad (5)$$

The final sintering stage is defined by a declining rate of densification and an accelerating rate of grain coarsening [61,64]. Sintering is much slower in the final stage since curvature gradients and surface energy are significantly reduced. According to Figure 2-13 [6], the pores forming a network during the intermediate sintering stage become spherical, isolated, and separated from the grain boundaries as the process moves forward. Densification is hindered by the coarsening of the pores and grains [6]. Rapid grain growth tends to strand pores away from grain boundaries, leading to delayed densification and residual porosity.

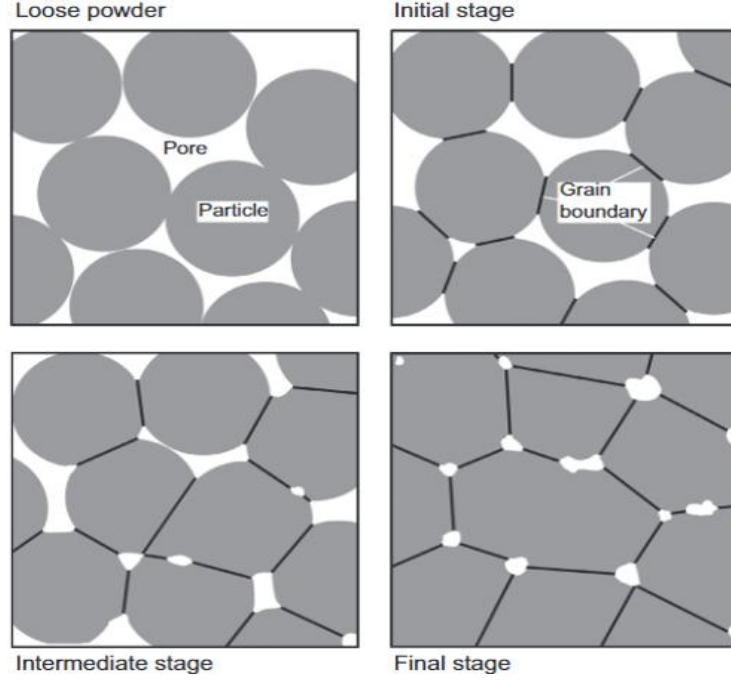


Figure 2-13 Porosity configuration during sintering stages [6].

In contrast, high pore mobility preserves the pore-boundary structure, quickly converting to full density. The most useful pores are the smallest, frequently moving by surface diffusion or evaporation-condensation [6,68]. However, grain boundary mobility is usually greater than pore mobility, causing pore drag and slowing grain growth during the last sintering stage. This holds as long as the pores remain attached to the grain boundaries. Once the grain boundaries separate from the pores, the grain grows quickly. Pore attachment to the boundaries is achieved by successful sintering, decelerating grain growth, or increasing pore mobility [6].

2.5 Master Sintering Curve

The master sintering curve (MSC) is a promising approach to defining and forecasting sintering behavior. Hansen et al. [69] developed a combined-stage sintering model by studying the mass-transport mechanisms during sintering. This model relates the densification rate, grain boundary, and volume diffusion, as shown in Eq. 6:

$$\frac{dL}{Ldt} = \frac{\gamma\Omega}{kT} \left(\frac{\Gamma_v D_v}{G^3} + \frac{\Gamma_b D_b}{G^4} \right), \quad (6)$$

Where L is the length, t is the time duration, Ω is the atomic volume, γ is the surface energy, T is the temperature, k is the Boltzmann's constant, Γ is the lumped scaling factor (the density-

dependent geometric terms), D is the diffusivity factor, and G is the grain size. The subscripts v and b are volume diffusion and grain boundary diffusion factors, respectively.

Su and Johnson [70] rearranged the combined-stage sintering equation by placing all the density-dependent material characteristics on one side and the known process-dependent factors, except Q , the apparent activation energy, on the right side. The modified equation is shown in Eq. 7.

$$\frac{K}{\Omega\gamma D_0} \int_{p_0}^P \frac{G(p)^n}{3P\Gamma(P)} = \int_0^t \frac{1}{T} \exp\left(-\frac{Q}{RT}\right), \quad (7)$$

The right-hand side of Eq. 7 can be replaced by the term Θ defining the work of sintering, as shown in Eq. 8:

$$\Theta = \int_0^t \frac{1}{T} \exp\left(-\frac{Q}{RT}\right). \quad (8)$$

The value for apparent activation energy can be determined through numerical analysis by assuming an initial value from literature and using many iterations [71]. The norm of the least squares residual method is used to identify the best value for Q through iteration, as shown in Eq. 9:

$$\min(x^2) = \sum_{i=1}^n \frac{\rho_{measured,i} - \rho_{cal,i}}{\rho_{measured,i}}. \quad (9)$$

In Eq.9, $\rho_{measured}$ refers to the relative density observed experimentally, and ρ_{calc} is the value anticipated by the combined stage sintering model. The MSC can compare the density values of various powders during the sintering process and forecast the final density of sintered products. Based on previous works [71,72], a sigmoidal function best describes the relationship between fractional densities and sintering work, as shown in Eq.10.

$$\rho = a + \frac{1-a}{1 + \exp\left(-\frac{(\ln\theta - b)}{c}\right)}. \quad (10)$$

The green part's relative density is represented by a constant a . The material affects constants b and c .

CHAPTER 3 LITERATURE REVIEW ON SINTERING OF BJ STAINLESS STEELS

3.1 Sintering of stainless steel 316L

3.1.1 An introduction to 316L

Stainless steel 316L is widely used in industrial applications, including the manufacture of equipment, tools, electronics, and automotive parts [73,74]. This material is often used for research and industrial applications in BJ, metal injection molding (MIM), and powder metallurgy (PM) due to its exceptional mechanical properties, excellent corrosion resistance, and weldability [55]. The constituent element contents of austenitic stainless steel 316L for the powdered and wrought states are provided in Table 3.1 [75,76]. Chromium (Cr), nickel (Ni), manganese (Mn), and molybdenum (Mo) are considered the main constituents and significantly affect the mechanical and corrosion properties. The role of chromium is vital because it hardens the steel and increases its corrosion resistance, especially at high temperatures [77]. Nickel stabilizes the austenitic structure at room temperature, improving the ductility, weldability, and high-temperature properties of 316L stainless steel [78]. Molybdenum adds corrosion resistance and high-temperature strength [78]. Manganese is added to steel to improve heat-working properties and increase strength, toughness, and hardening [78]. From a microstructure perspective, 316L stainless steel is an austenitic steel with a small amount of ferrite due to a high amount of nickel (Ni) and a small amount of manganese (Mn) acting as austenite phase stabilizer at room temperature [74]. Stainless steel 316L also has a low amount of carbon (C) (around 0.03%) to increase ductility and reduce sensitization, chromium carbide formation, and intergranular corrosion [74]. The phase stability of 316L stainless steel can be understood using the Fe–Cr–Ni phase diagram. As shown in Figure 3-1, increasing nickel content stabilizes the γ -austenite phase, while α -ferrite is stable at lower nickel concentrations and higher temperatures. At elevated temperatures, phase transformations and the possible formation of δ Ferrite influences diffusion behavior and sintering kinetics.

Table 3.1 Stainless steel 316 chemical composition as a wrought [75] and Gas atomization (GA) Powder [76].

Composition %	Fe	Cr	Ni	Mo	Mn	Si	C	O	S
Powder (GA)	Bal.	16-17	11-12	2-3	< 1.5	< 1.0	< 0.013	< 0.10	< 0.01
Wrought	Bal.	16-18	10-14	2-3	< 2.0	< 1.0	< 0.03	< 0.14	< 0.004

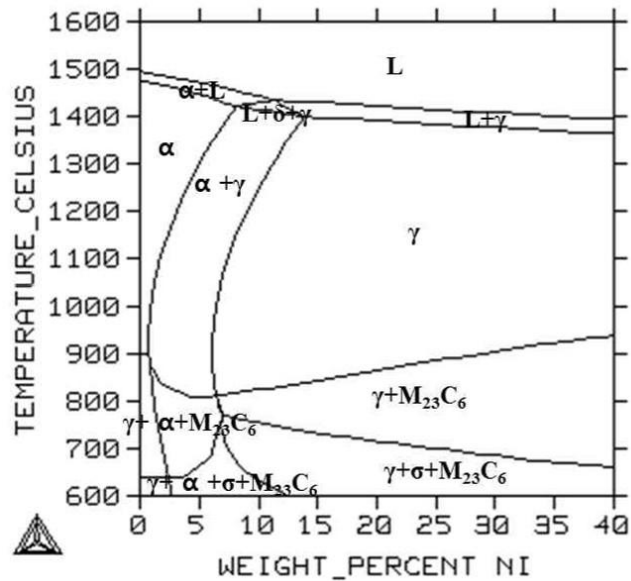


Figure 3-1 Fe–Cr–Ni phase diagram showing phase stability (γ -austenite and δ ferrites) for 316L [74].

The manufacturing method affects the final microstructure of 316L stainless steel. Figure 3-2 demonstrates the microstructure of cast and BJ-sintered stainless steels. For cast 316L (Figure 3-2 a), the phases such as carbides, δ ferrites, and γ austenite are observed [74]. Conversely, for BJ-sintered 316L (Figure 3-2 b), the microstructure (sintered at 1360-1380 °C) includes additional features such as porosities and twin boundaries [20].

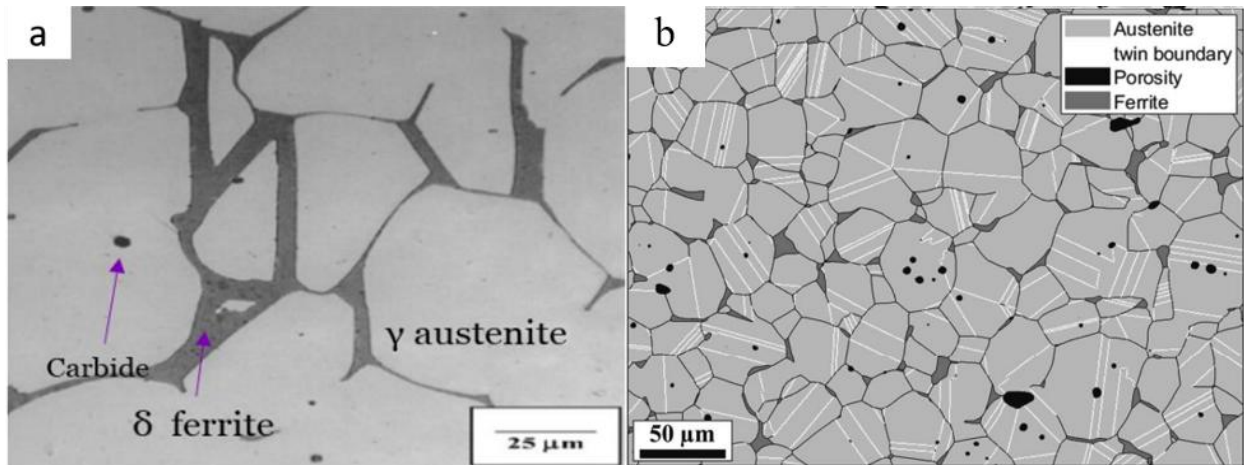


Figure 3-2 Microstructure of a) cast and b) BJ-sintered SS 316L [20,74].

3.1.2 Powder characterization

In binder-jetted SS 316L parts, particle size distribution (PSD) substantially influences the powder bed packing density. A wider PSD provides higher packing densities than a narrower PSD [33]. Multimodal powders can boost powder density since coarse powders improve flowability. In contrast, the fine powders fill the spaces left by the larger particles [53]. A "bimodal" PSD is obtained when two distinct powder particle sizes, or "modes," are combined at a specific size and volume ratio [79]. Literature [25,44] showed that a ratio of 7:1 (coarse to fine) caused a real improvement in packing density in 316 printed parts.

The impact of particle size, morphology, sintering temperature, and time on the final characteristics of sintered 316L parts was studied by Verlee et al. [80]. Table 3.2 shows the green density, particle means diameter, and sintered (final) density for six different powder sizes at 1395 °C. The particle size has a pronounced effect on the final density. The final densities produced by smaller particle sizes lower than 45 μm (>96%) are significantly higher than larger particle sizes above 45 μm (<70%). The increased densification for smaller particles is attributed to higher surface energies than larger particles. Furthermore, smaller pores between finer particles cause earlier sintering compaction. Hence, smaller powders can produce higher densities at lower sintering temperatures [80].

Table 3.2: Effect of particle sizes and shape on the sintered density of BJ SS 316L at 1395 °C [80].

Powder Size (D90)	Green Density (%)	Particle mean diameter (μm)	Sintered (final) density (%)
22 μm spherical	58.6	13	96.1
31 μm spherical	57.6	17	97.4
45 μm (non-spherical)	34.6	27	60.9
53 μm spherical	54.8	38	66.3
90 μm (non-spherical)	29.0	64	46.7
90 μm spherical	55.5	73	64.6

3.1.3 Temperature and time effect on the sintering behavior

According to the sintering theory [81], the ultimate density will rise with increasing temperature until full densification is reached. Juan [77] investigated the effects of temperature and time on sintered density, porosity shape, and pore distribution. Figure 3-3 shows that smaller particles reach higher densities at lower temperatures. For the powder with a particle size of 15 μm , the curve begins to drop at a temperature of 1380 $^{\circ}\text{C}$, showing that increasing the temperature beyond 1356 $^{\circ}\text{C}$ has little effect on densification [82].

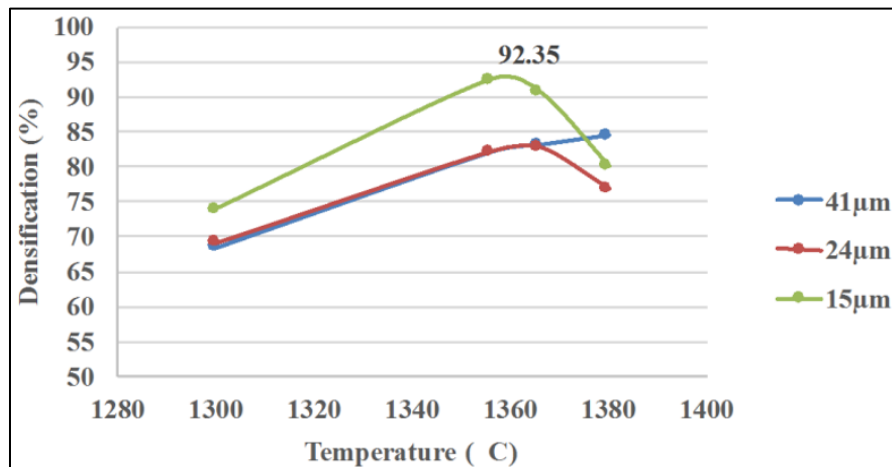


Figure 3-3 Densification (%) versus temperature ($^{\circ}\text{C}$) of BJ SS316L for powders with three different particle sizes [82].

The cause of decreasing density with increasing temperature was explained by the microstructure evolution in Figure 3-4 (a-d) [82]. Figure 3-4(a) shows that many large pores are connected to the grain boundary and triple grain junction. Grain boundary diffusion was the predominant mechanism when the porosity was connected to the grain boundaries. Although Figure 3-4 (b) shows pore-boundary separation, most pores were tiny and isolated within the grains. This indicated the final step of sintering and densification. As shown in Figure 3-4 (d), the pore grew larger and emerged on the grain boundary again when the temperature was raised further. The size of the pores increased while the number of pores remained unchanged [82]. Another theory for the decline in sintered density is that isolated pores' capillary pressures reached a pressure equilibrium with trapped gas, stopping the pores from contracting [67,82].

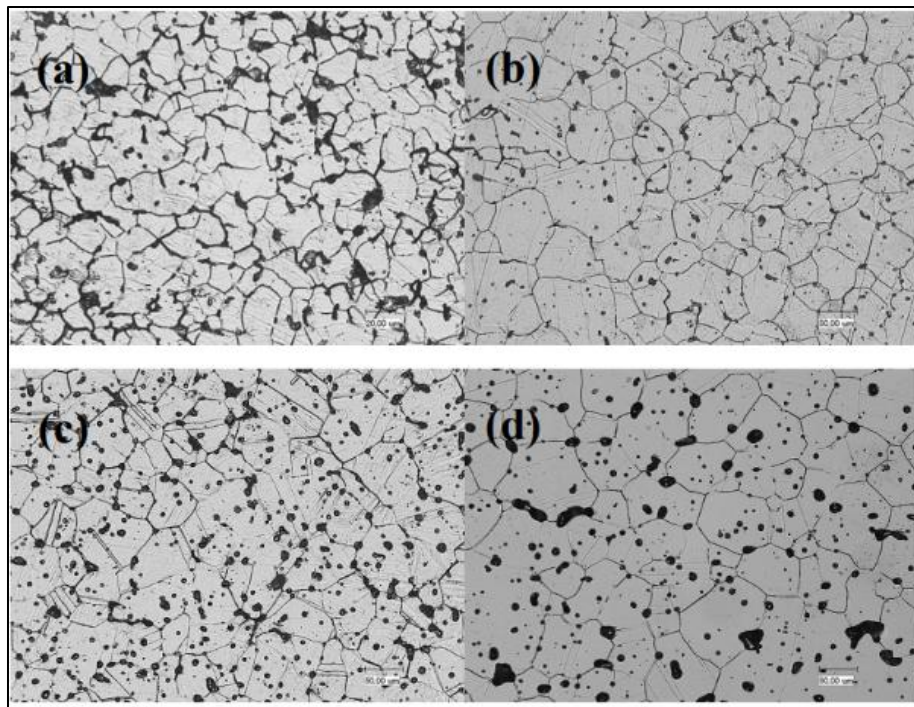


Figure 3-4 Microstructure of BJ SS316L sintered at various temperatures, with a powder size of 15 μ m, including (a) 1300 °C, (b) 1356 °C, (c) 1366 °C, and (d) 1380 °C for 90 mins under argon atmosphere [82].

Juan [82] also studied how final densities were affected by sintering holding time. As shown in Figure 3-5, samples were kept at 1300 °C, 1356 °C, and 1380 °C for 90, 360, and 1440 minutes, respectively. The findings showed that for samples sintered at 1300°C, increasing the time increased the final density. The maximum density was achieved for the sample sintered at 1356 °C

for 360 min. After this point, increasing the holding time did not improve the sintered density. This behavior was attributed to a minor change in the pore number and larger pore sizes [82].

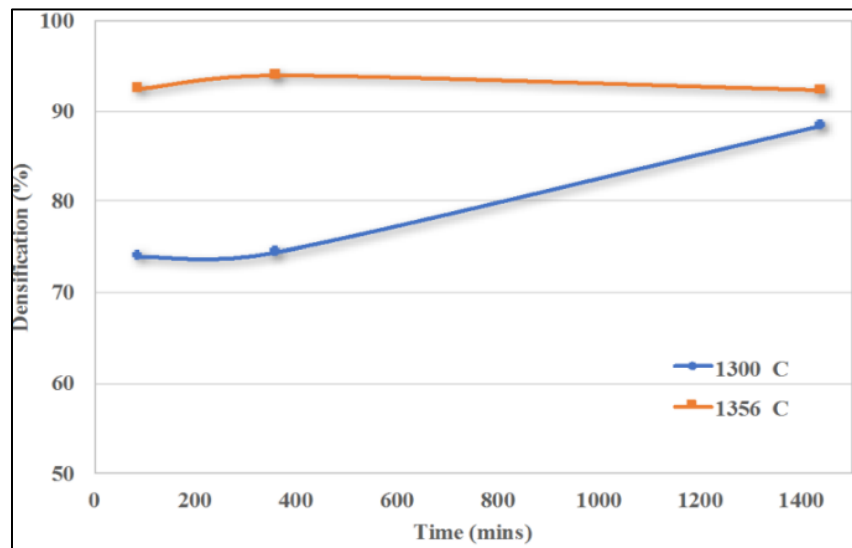


Figure 3-5 Density versus time at two sintering temperatures in BJ SS 316L with a powder size of 15 μ m [82].

3.1.4 Anisotropy shrinkage during sintering

Managing the final BJ part dimensions is challenging due to anisotropic shrinkage during sintering. Printed parts shrink during sintering to eliminate pores brought on by printing. Powder properties and the sintering step are significant aspects of binder jetting in which PSD, powder morphology, and sintering temperature can significantly affect the final shrinkage and density [83]. Powder-packing density determines the final product's sintering shrinkage and mechanical strength. Particle size distribution (PSD) substantially influences the powder bed's packing density. A wider PSD provides more packing densities than a narrower PSD. As a result, in binder jetting, bimodal powders, because of their greater packing factors, have the potential to increase sintered density (by around 4%) while decreasing sintering shrinkage (by about 7%) [84]. Moreover, the packing density, sintering shrinkage, and to some extent, the powder's flowability are all influenced by the shape of the powder particles. Figure 3-6, for example, illustrates how particle shape affects stainless steel shrinkage level. The largest shrinkage belonged to the powders with irregular shapes [85].

Rios et al. [86] performed sintering dilatometry experiments to investigate the sintering behavior of SS 316L BJ parts in different directions of X (recoating direction), Y (printhead movement

direction), and Z (build direction). The first part of the paper determined how the debinding procedure affected the dimensional evolution during sintering. The dilatometry curve (Figure 3-6) showed that the green sample during the debinding step exhibited both shrinkage and expansion behavior. This behavior can be related to the particle rearrangement that occurs during debinding, particularly at temperatures when the primary chemical constituents of the binder are broken down [86].

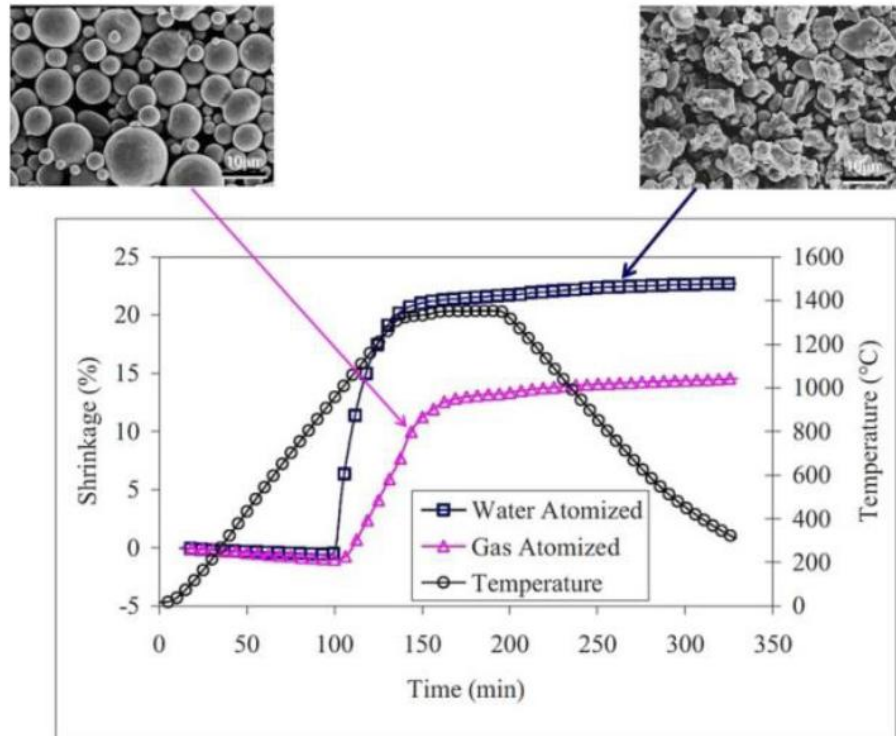


Figure 3-6 The influence of powder shape on sintering shrinkage of BJ SS 316L [85].

During the debinding stage, shrinkages along the three axes are almost identical (Figure 3-7). The noteworthy point is that the dimension changes along the building direction (z-axis) were the lowest. However, with a temperature of more than 800 °C, the shrinkage rate started to accelerate along the Z-axis, and anisotropy developed afterward. The shrinkage seen after debinding (0.4%) is negligible compared to the overall shrinkage. These findings demonstrated that shrinkage anisotropy is not directly caused by binder removal and potential particle rearrangement during the debinding stage [86].

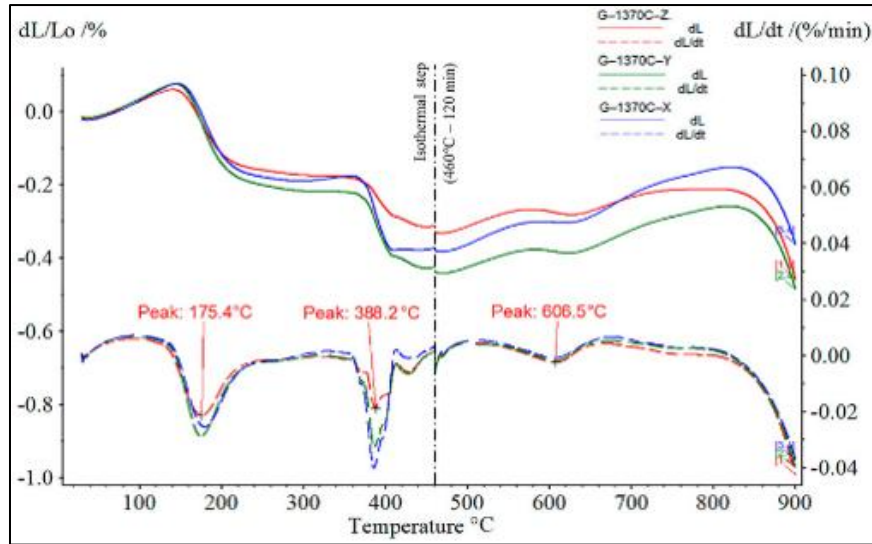


Figure 3-7 Dimensional changes in the debinding step in BJ SS 316 [86].

In the second part, sintering dilatometry experiments described the sintering behavior of 316L BJ in different axes of the powder bed at 1370 °C. The dilatometry curve during sintering is split into two sections depending on the onset of sintering temperature. Until 942 °C, below the onset of sintering temperature, samples in three axes displayed positive and similar values related to expansion. Above this temperature, parts started to shrink, and compared to the two other axes, the build direction axis underwent greater sintering shrinkages (Figure 3-8) [86]. According to Figure 3-8, due to different diffusion mechanisms, such as grain boundary and surface diffusion, the shrinkage rate quickly grew to a maximum relative value of 0.2 % [86].

The shrinkage rate gradually dropped to 0.12 % because of grain growth and the reduced diffusion rate. At 1370 °C, the shrinkage rate significantly increased by 38 %/min. This increase can be attributed to the transformation of austenite into ferrite, which improves the diffusion rate and sintering activity [86,87]. Finally, the shrinkage rate dropped to zero during the isothermal step. This decrease may have been brought about by the isolation of pores and a reduction in free surface area during grain growth [61,88–90].

To date, very few studies have investigated anisotropy shrinkage during sintering. The presented works only focus on 316L stainless steel at two different temperatures. Moreover, the effects of important parameters, such as activation energy, sintering viscosity, and green density, have not been studied.

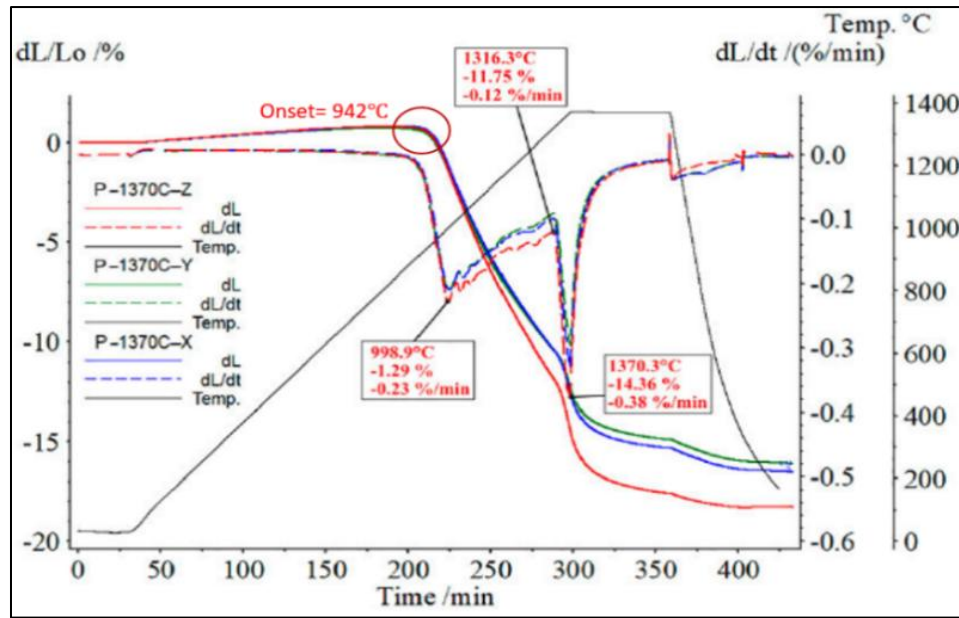


Figure 3-8 Dimensional changes along different axes during sintering at 1370°C for pre-sintered SS 316 BJ [86].

3.1.5 Microstructure

Rios et al. [87] concluded that most anisotropy in sintering shrinkage arises after phase transformation. They examined the microstructure of the BJ-fabricated components as they were being sintered. The EBSD maps made from the XZ cross-sections of samples sintered at 1300°C and 1370°C, respectively, are shown in Figure 3-9 (a) and (b). The common phases at two temperatures include FCC austenite (γ), grain boundary, twin boundary, and porosity. But the difference is that when the sintering temperature reaches 1370 °C, the BCC ferrite phase (δ -ferrite) is replaced by larger pores. It was confirmed that shrinkage increased excessively at temperatures above 1340°C by the presence of the δ -ferrite phase surrounding the austenite phase at the intersection between grains [87,91–93]. Since there is no comprehensive information about the microscopic structure of BJ samples during anisotropic sintering and no studies linking the diffusion mechanisms to the anisotropic sintering, these phenomena need to be investigated more carefully.

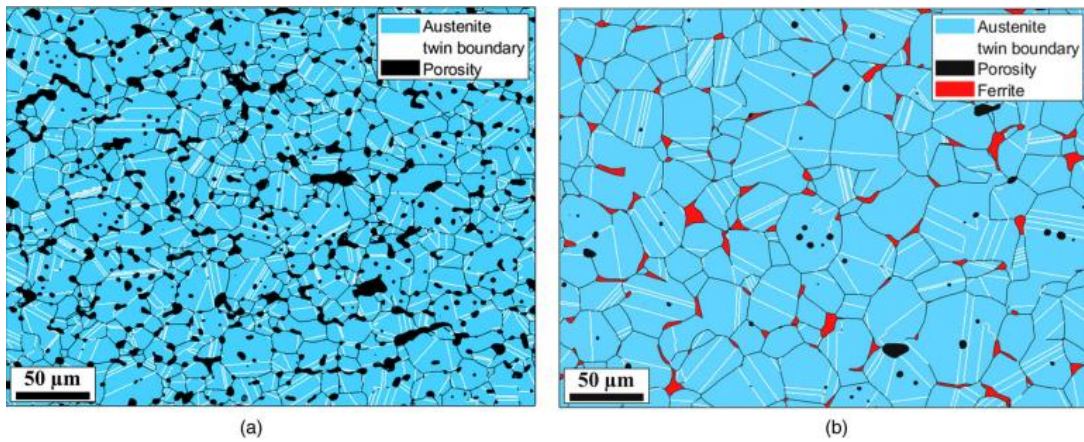


Figure 3-9 EBSD maps of BJ SS 316 L sintered at 1300°C and 1370°C [87].

3.1.6 Mechanical properties

Final density, grain size, and pore size influence binder jetting mechanical properties. Verlee et al. [80] showed that the samples sintered at higher temperatures reached higher strength and elongation due to lower percentages of porosity and higher final densities. Since pores act as high-stress concentration sites that create and propagate cracks, reducing their content can significantly enhance the mechanical characteristics of BJ parts. Table 3.3 shows the relation between the final sintered density and mechanical characteristics (elongation and tensile strength) for two powder sizes [90].

Table 3.3: The final density and mechanical properties of BJ SS 316 for two different powder sizes [90].

Powder	Sintering temp./°C	Density/%	Max. strength/MPa	Elongation/%
		±0.1%	±6%	±5%
31 µm	1255	81.0	309	21.3
	1335	85.2	388	35.5
	1365	90.9	437	52.1
	1395	98.0	518	61.9
20–53 µm	1415	81.8	243	25.4
	1432	88.2	310	29.9

The sintering atmosphere greatly influences the microstructure and final properties of BJ materials. It has been claimed that 316L sintered samples in a vacuum had higher elongation and strength than those in gas atmospheres like hydrogen and argon [20]. One of the negative points regarding gas atmosphere sintering is that gas atoms like hydrogen atoms diffuse into the matrix and pores,

which prevents the elimination of porosity in the final stage of sintering. This limits densification and may promote hydrogen embrittlement and ultimately result in loss of ductility [20,94].

Table 3.4: Comparison between the mechanical properties of 316L produced by BJ and conventional manufacturing [20].

Material	Relative porosity (%)	Sintering profile	Yield strength (MPa)	UTS (MPa)	Elongation to fracture (%)	Charpy impact, room temperature (J)	Hardness (HV)-XY plane	Hardness (HV)-Z direction
BJAM 316 L (this study)	1.9 ± 0.1	2 h, 1380 °C, vacuum	191 ± 8	544 ± 25	73 ± 2.8	62 ± 7	138 ± 4	143 ± 6
BJAM 316 L[33]	$\sim 3.7 - 5.6$	2 h, 1380 °C, H ₂	$\sim 189-198$	$\sim 534 - 558$	$\sim 64 - 80$	-	-	-
BJAM 316 L[19]	~ 2.6	1.5 h, 1395 °C, H ₂	-	518 ± 6	61.9 ± 5	-	-	-
BJAM 316 L[22]	$\sim 1.7-4.2$	3 h, 1360 °C, vacuum	~ 176	~ 535	~ 49	-	~ 148	-
Conventional 316 L-HRP[33]	-		273 ± 37	622 ± 6	65 ± 10	-	~ 385	-

Table 3.4 compares the mechanical characteristics of SS 316L BJ sintered in vacuum and H₂ with a hot-rolled plate of 316L [20]. The BJ tensile test results showed high ductility (>73 %) for samples sintered under a vacuum atmosphere. The authors reported that the excellent ductility of BJ samples sintered in a vacuum is due to the shape and size of the porosity [20]. The small and rounded porosity in the 316L BJ vacuum-sintered sample provides the lowest stress concentration factor and prevents crack development. However, the BJ tensile properties (yield and ultimate tensile strength) were lower than those of hot-rolled 316L by 42% and 15%, respectively. The porosity percentage of two or more BJ samples can be used to explain this difference. Localized stress is amplified by pores, which causes plastic deformation to begin at low-stress levels [20,94]. Another factor contributing to the lower tensile properties of BJ samples was their larger grain sizes, which are inherent to the process [95].

Kumar et al. [96] also investigated the high-cycle fatigue (HCF) behavior of 316L austenitic stainless steel fabricated by three different methods of binder jet printing, selective laser melting, and conventional manufacturing (CM). Their results showed that despite having many pores, the fatigue strength (σ_f) of the BJ specimens was surprisingly close to that of the CM alloy. On the other hand, the SLM specimens exhibited lesser ductility and fatigue strength while having higher yield strength and UTS than the CM alloy. The SLM specimen's fatigue strength is noticeably lower at 101 MPa and may be increased to roughly 250 MPa by lowering the porosity level. Unexpectedly, BJ SS 316L has a fatigue strength of 250 MPa despite having relative porosity levels

between 3.7 % and 5.6 %. According to their results, they concluded that the porosity of the BJ alloys has no negative effects on ductility or fatigue strength. Because of the planar slip regime that predominates during the initial stages of plastic deformation in BJ alloys and a combination of other microstructure variables, small cracks are stopped from forming at the pore corners [96]. Interrupted tensile tests were performed on BJ specimens to determine how the slip plane affects fracture initiation. As shown in Figure 3-10a, the experiments were stopped for microstructural analysis by SEM at three stress values of 250, 270, and 400 MPa. In Figure 3-10 b, only the planar slip can be seen under a stress of 250 MPa (a strain of 1.36%). As shown in Figure 3-10 c, the higher the stress ($\epsilon = 2.14\%$ and $S = 270$ MPa), the more microcracks formed around the pores. These cracks seem to be aligned with the slip lines. As shown in Figure 3-10 d, the duplex slip (initially appearing around the pores) expands as the stress (S) increases until 400 MPa. In this stage, the cracks formed near the pores were not aligned with the slip lines because the duplex slip lines originated at an obtuse angle to the plane slip lines. As a result, any cracks in the pores' corners were blocked [96,97].

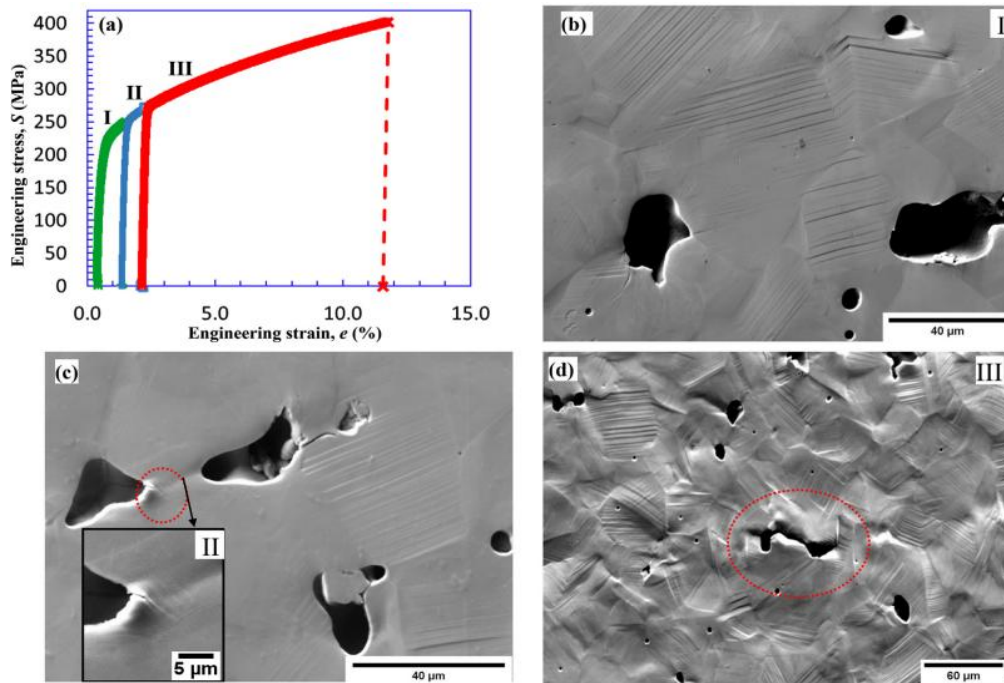


Figure 3-10 (a) Engineering stress against strain in BJ SS 316 samples. The effect of different strain levels on the microstructure: (b) $\epsilon = 1.36\%$, (c) $\epsilon = 2.14\%$, and (d) $\epsilon = 11.56\%$ [96].

HSR (high strain rate) deformation is important since rapid loading can significantly modify the mechanical response and failure mode relative to quasi-static behavior; therefore, dynamic data are required for robust design across relevant strain-rate and temperature ranges [98,99].

The split Hopkinson pressure bar (SHPB) (Figure 3-11) has been shown in is a dynamic testing method used to measure a material's stress–strain response under rapid loading [99]. In this setup, a small specimen is sandwiched between two long elastic bars (incident and transmission bars) [99,100]. A striker impacts the incident bar and generates a stress wave that travels into the specimen; part of this wave is reflected back into the incident bar and part is transmitted through the specimen into the transmission bar. Strain gauges mounted on the bars record the incident, reflected, and transmitted waves, and these signals are converted using one-dimensional elastic wave relation into the specimen's strain rate, strain, and stress, yielding a dynamic stress–strain curve.

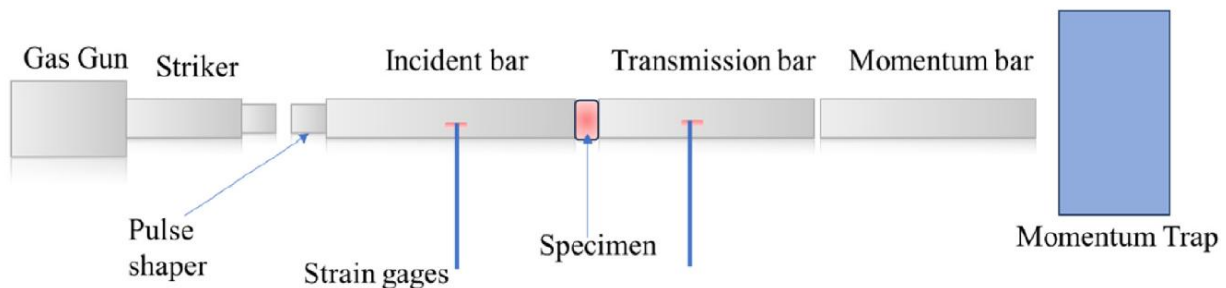


Figure 3-11 Schematic of the split Hopkinson pressure bar (SHPB) setup used for dynamic compression testing [99].

Lee et al. [101,102] studied wrought/biomedical 316L using SHPB compression over a wide range of strain rates and temperatures and reported that the flow stress and work hardening are strongly influenced by both variables: the material strengthens with increasing strain rate and softens with increasing temperature, and the apparent rate sensitivity changes with strain.

For additively manufactured 316L, Li et al. [103] tested LENS-316L in SHPB compression at 3000–6000 s^{-1} and 20–620 $^{\circ}\text{C}$ and showed that AM microstructure can modify the dynamic response and localization behavior. They reported that dislocation strengthening is dominant, and they observed increased deformation twinning as strain rate increased. At higher rates ($\approx 5000 \text{ s}^{-1}$), they reported adiabatic shear band formation, with shear-band width decreasing as strain rate

increased; they also proposed a modified Johnson–Cook model that better captures coupled strain-rate and temperature effects during strain hardening.

3.2 Sintering of stainless steel 17-4 PH

3.2.1 Composition

The 17-4 PH SS is a martensitic stainless steel that is precipitation-hardened (PH) by the addition of copper (Cu) [104]. This material generally consists of 0.3% niobium, 4% nickel, 4% copper, and 17% chromium [105]. Adding chromium (Cr) and nickel (Ni) improves the steel's corrosion resistance, strength, hardness, and ductility. Copper promotes the formation of copper-rich precipitates during heat treatment, which act as nucleation sites for forming the martensite phase and Cu-enriched particles. The precipitation phases allow SS17-4 PH to achieve a higher strength and hardness level. Additionally, adding Niobium (Nb) can suppress the creation of chromium carbides in 17-4 PH stainless steel, reducing corrosion resistance and toughness [104]. Table 3.5 shows the nominal compositions of wrought and powder SS 17-4 PH.

Table 3.5: Nominal composition of 17-4PH for wrought and powder alloys (wt. %) [105].

Composition	Cr	Ni	Cu	Si	Mn	Nb+Ta	C
Wrought	15.5	4.5	3.5	0.5	0.4	0.3	0.04
Powder	16.5	4.0	4.0	1 Max	1 Max	0.3	0.07

The carbon content in 17-4 PH stainless steel is typically very low (≈ 0.03 – 0.07 wt. %) [105]. According to the Fe–C phase diagram (Figure 3-12), this composition lies in the low-carbon region, where austenite (γ) is stable at high temperatures and transforms to martensite upon cooling [106]. The low carbon content limits carbide formation and promotes martensitic transformation, while the presence of δ -ferrite can also be retained due to the high chromium content. Therefore, the final microstructure of 17-4 PH consists mainly of lath martensite with a small fraction of δ -ferrite, consistent with the observations Figure 3-13, which depicts the microstructure of SS 17-4 PH as a wrought. This consists of parallel bands of lath martensite as the substrate and ferrite phase elongated in the primary austenite grain boundaries [107].

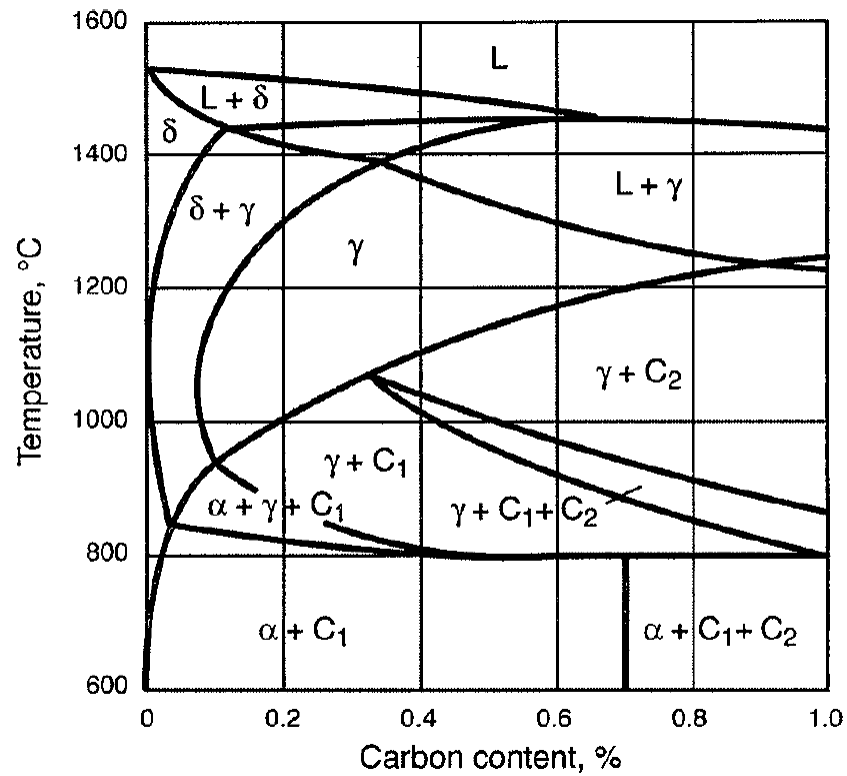


Figure 3-12 Fe–C phase diagram illustrating phase stability relevant to 17-4 PH stainless steel [106].

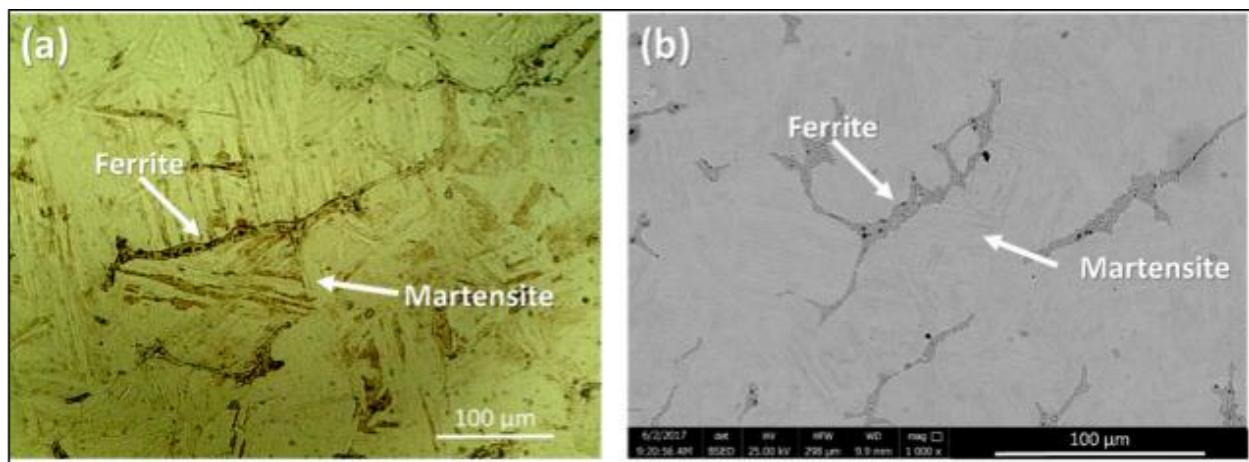


Figure 3-13 Microstructure of the wrought 17-4 PH SS; (a) optical and (b) SEM [107].

The EDX analysis of the sample's constituents, including the ferrite and martensite phases, are shown in Table 3.6 [107]. According to the results, the δ -ferrite phase contains a higher

concentration of Cr in spherical precipitates and a few unusually formed Cu-enriched particles (CRPs). The martensite phase, in contrast, displayed an even distribution of Cr and Cu elements [107].

Table 3.6: Chemical composition of wrought 17-4 PH SS (wt. %) [107].

Phase %	Cr	Ni	Cu	Si	Mn	Fe
Ferrite	22.3	1.6	1.2	0.4	0.1	Bal.
Martensite	16.5	3.7	2.9	0.3	0.1	Bal.

3.2.2 Heat treatment

The standard heat treatment for SS17-4 PH, including solution annealing and aging, is summarized in Table 3.7. Solution heat treatment involves heating the material to 1040 ± 15 °C for a certain time to dissolve the alloying elements in the austenitic matrix. Quenching forms a soft martensitic structure supersaturated with copper [108]. The aging treatment for SS 17-4 PH happens at a temperature between 482 °C and 760 °C for 1 to 4 hours. Coherent copper-rich precipitates form during aging, resulting in precipitation hardening of the material. These precipitates become incoherent FCC Cu precipitates after long aging [108]. It has also been reported that M23C6-type particles form in 17-4 PH steel during aging [109]. An aging temperature above 580 °C may cause material softening, improved ductility, and larger fracture toughness due to the reversion of martensite to austenite [109].

Table 3.7: Technical heat treatment for SS 17-4 PH [108].

Condition	Solution Heat Treatment	Precipitation Treatment		Yield Strength (MPa)	Tensile Strength (MPa)	Hardness (RC)
		Temperature (°C)	Time (hour)			
H900	Solution heat-treatment at 1040°C-1066°C (Condition A)	482 $\pm$ 5	1	1170	1310	40-47
H925		496 $\pm$ 5	4	1070	1170	38-45
H1025		552 $\pm$ 5	4	1000	1070	35-42
H1075		579 $\pm$ 5	4	860	1000	33-39
H1100		593 $\pm$ 5	4	790	965	32-38
H1150		621 $\pm$ 5	4	725	930	28-37
H1400		760 $\pm$ 5	4	515	790	26-36

3.2.3 Powder characteristics

There is little information about BJ SS 17-4 PH powder characteristics in the literature. However, since the powder properties in metal injection molding (MIM) are similar to BJ, this section presents the literature for both methods. In the case of 17-4 PH stainless steel, pre-alloyed powder atomized with gas or water is preferred for MIM and BJ [105]. The spherical powders produced by gas-atomization (Figure 3-14 a) provide a desirable high packing density and acceptable flow characteristics provided sticking to tiny satellite particles is prevented. Water-atomized powders with irregular shapes (Figure 3-14 b) have higher oxygen content due to the exposure of molten metal to high-pressure water. However, the oxygen in these powders reacts with the remaining carbon in the binder, reducing the sensitivity to the debinding parameters [110]. Average gas atomized powder characteristics of 17-4 PH are summarized in Table 3.8.

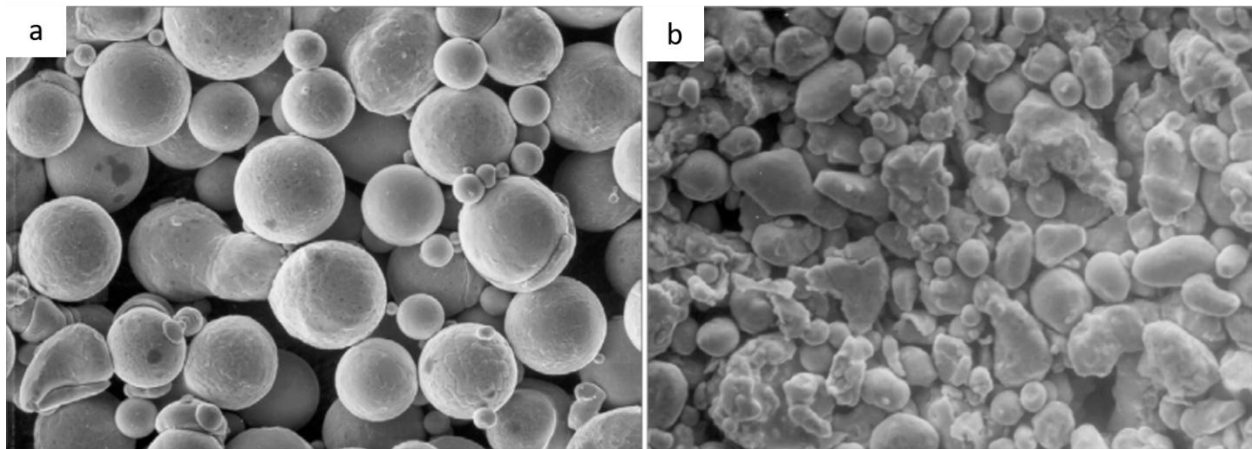


Figure 3-14 Powder morphology of 17-4 PH SS: (a) spherical gas atomized and (b) water-atomized powders [105].

Table 3.8: Typical gas atomized powder characteristics of SS 17-4 PH [105].

Pycnometer Density	4.8 g/cm ³
Tap density	4.3 g/cm ³
D10 Particle Size	5 μm
D50 Particle Size	11 μm
D90 Particle Size	24 μm
Carbon Content	0.05%
Oxygen Content	0.3%

3.2.4 Sintering parameter effects

Sung et al. [111] studied the effect of sintering temperature on shrinkage and the final relative density in MIM SS 17-4 PH. Figure 3-15 shows that the relative density of the samples has been adjusted from 61% to 99% by changing the sintering temperature from 900 °C to 1350 °C. As the sintering temperature was raised, the pores of the sintered specimens appeared to become more rounded and isolated [111]. Huber et al. [112] investigated how the sintering temperature and time affected the densification, mechanical properties, and microstructural characteristics of 17-4 PH fabricated by BJ. Printing was conducted on an Innovent ExOne using a gas-atomized powder of 45 μm and an organic aqueous binder. Following printing, the green samples were cured for six hours at 180 °C. All components underwent debinding and hydrogen atmosphere sintering.

For the sintered 17-4 PH, the microstructure from the middle to the edge of the sample was slightly different due to the heat transfer conditions (Figure 3-16). According to Hubert et al. [112], increasing the sintering temperature causes martensite elongation and δ -ferrite network formation. The effect of temperature on the microstructure is shown in Figure 3-17. The porosity decreased from 7% to 2% with increasing temperature from 1270 °C to 1360 °C while the pores became more isolated and equiaxed. However, the porosity increased again at 1360 °C, especially in the center of the sample. Porosity was generally higher in the middle than at the edge of the sample [112].

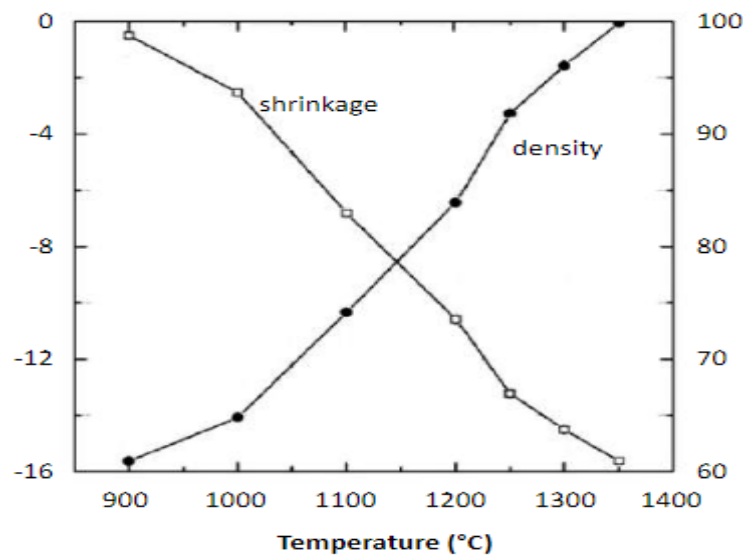


Figure 3-15 The effect of sintering temperature on linear shrinkage and relative density of MIM SS 17-4 PH [111].

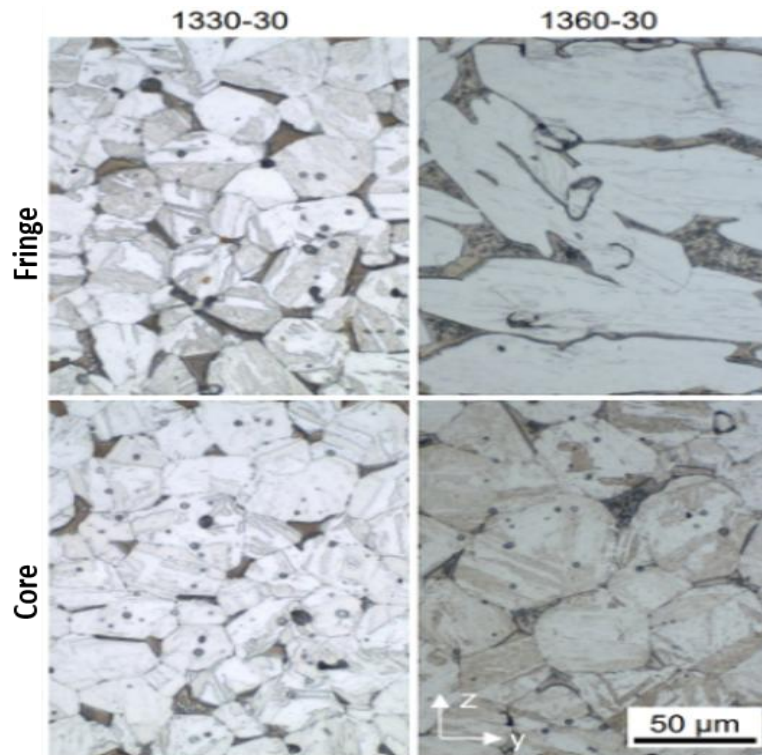


Figure 3-16 Optical microstructure of the middle (core) and edge (fringe) of the BJ SS17-4PH as-sintered condition [112].

In sintering of 17-4 PH, most densification occurs during heating; therefore, only a brief period of holding is needed [91]. According to the literature [105,112,113], the sintering time for this material is limited to 60-80 minutes to provide adequate densification and prevent negative grain growth.

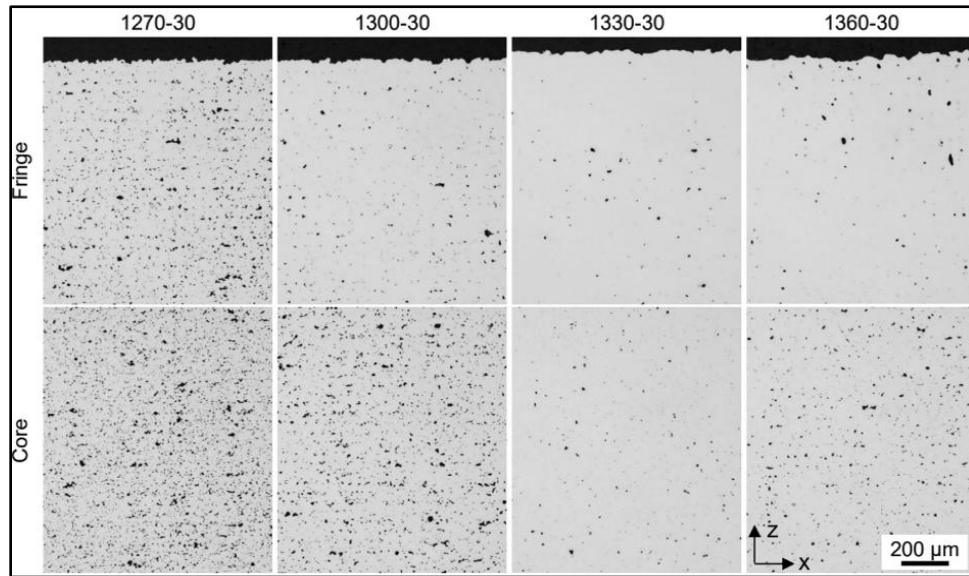


Figure 3-17 Optical micrographs showing the effect of sintering temperature on porosity in BJ 17-4 PH [112].

Typically, δ -ferrite forms at 1190–1220 °C during sintering for 17–4 PH [105]. The role of delta-ferrite (δ Fe) is mixed as it improves high-temperature sintering but can deteriorate the material's mechanical characteristics [105,110]. Retained δ -ferrite lowers the martensite content after heat treatment, decreasing the tensile strength. The volume fraction of δ Fe must be regulated to achieve optimal part properties. Previous works reported less than a 10% volume fraction to achieve sufficient UTS and elongation at break [105,110]. The δ Fe content depends on the carbon amount, sintering time, and temperature [105,110,112].

3.2.5 Mechanical properties

As mentioned in section 3.1.6, there is a linear connection between mechanical properties and sintered density [80,105]. Huber et al. [112] investigated the effect of sintering parameters on the mechanical properties of a BJ SS 17-4 PH sample. Figure 3-18 shows the effect of sintering temperature (Part I) and time (Part II) on the UTS (Figure 3-18 a), hardness (Figure 3-18 a), and ductility (Figure 3-18 b). Firstly, all mechanical characteristics were initially improved by increasing the sintering temperature (1270-1330 °C - Part I) and hold times (30-90 mins – Part II). For example, the highest UTS (100 MPa) and ductility (4%) were observed at 1300 °C and 90 min hold time, as shown in Figure 3-18 a and Figure 3-18 b Part II, respectively. According to the

authors, improved mechanical properties at high sintering temperatures and times are attributed to reduced porosity, as shown in Figure 3-17.

However, this behavior does not hold for different temperatures and time increases. Figure 3-18 shows that the mechanical properties degrade beyond a threshold temperature and time. For instance, considering Figure 3-18 Part I, as the temperature increases from 1330 °C to 1360 °C, the UTS decreases from 1050 MPa to 950 MPa, respectively. Similarly, as the hold time increases from 90 mins to 180 mins, the ductility decreases from 4% to 5% respectively. According to the literature [112–116] coarse martensite grains, high δ Fe content (>10% volume fraction), and large spherical holes in the microstructure can explain the strength loss at 1360 °C for 30 min.

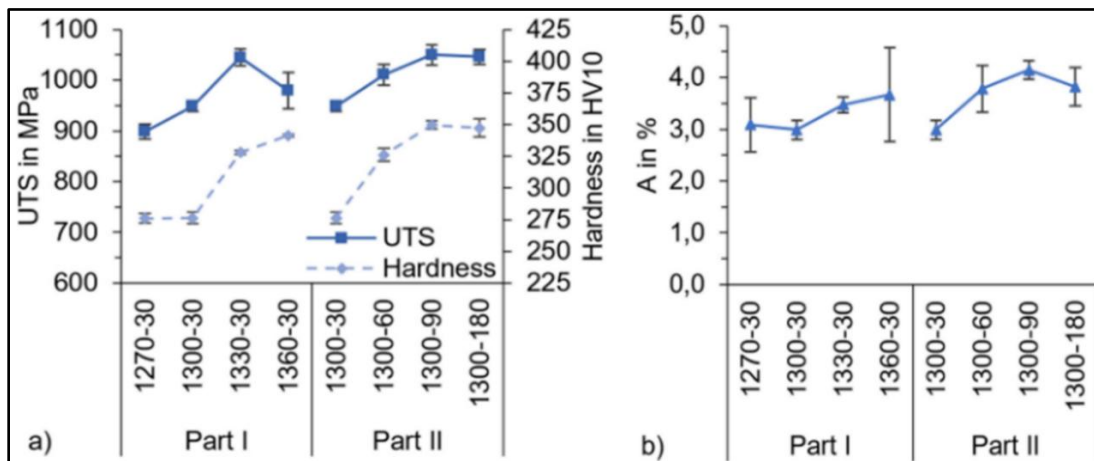


Figure 3-18 influence of sintering time and temperature on BJ SS 17-4 PH (a) UTS, hardness, and (b) elongation [112].

Irrinki H [117] compared the mechanical properties of several steel sheets, such as 17-4 PH and 316L, made by MIM and laser powder bed fusion (LPBF), with their wrought state. Figure 3-19a shows that the yield strength of MIM parts fabricated using 17-4 PH stainless steel samples was higher (750 ± 40 MPa) than the wrought (700 ± 30 MPa) and LPBF (550 ± 30 MPa) yield tensile strength. The SS 17-4 PH manufactured with the MIM process exhibited UTS values comparable to LPBF and wrought parts (Figure 3-19b).

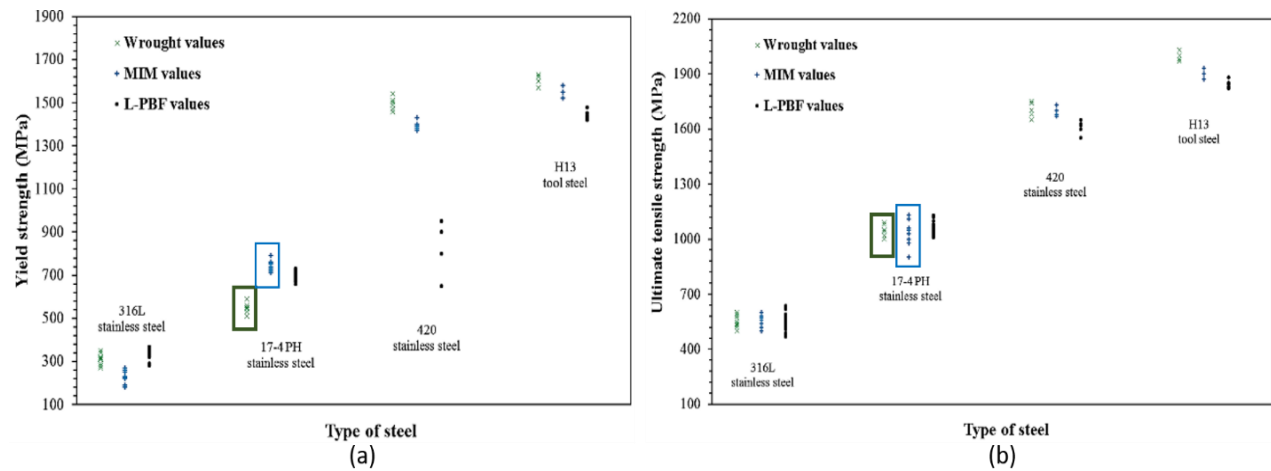


Figure 3-19 Comparison of yield strength and ultimate tensile strength of 17-4 PH wrought and 17-4PH fabricated by MIM and LPBF [117].

Elongation (%) values for LPBF, MIM, and wrought parts are shown in Figure 3-20. The 17-4 PH stainless steel elongation values produced by MIM fabrication were 12% less than the wrought and LPBF elongation values.

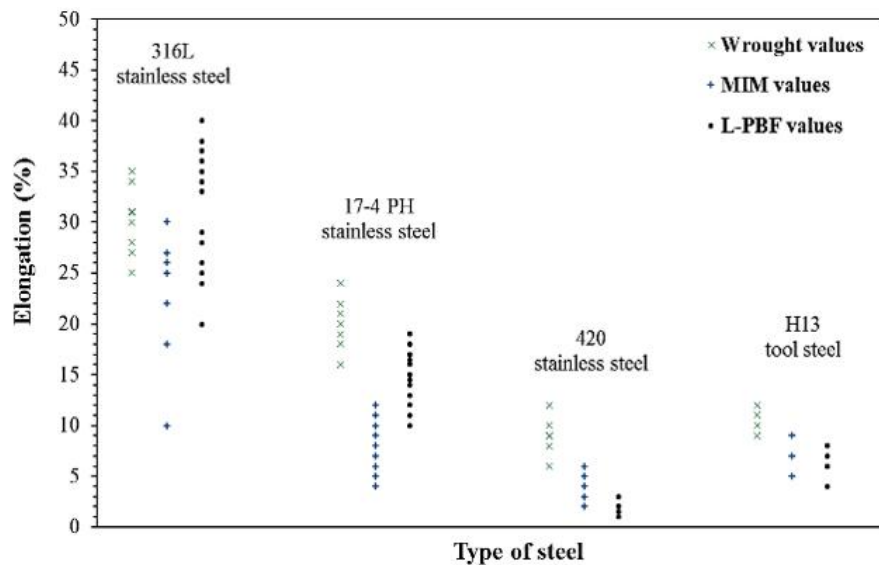


Figure 3-20 Elongation percentage of 17-4 PH wrought and 17-4PH fabricated by MIM and LPBF [117].

CHAPTER 4 SCIENTIFIC APPROACH

4.1 Critical evaluation of the literature review

The following knowledge gaps are identified based on the presented literature review on the BJ of two different stainless steel types, 316L (austenitic) and 17-4 PH (martensitic):

1. Sintering is a critical step in binder jetting (BJ), as it strongly influences porosity, microstructure, and the mechanical properties of the final parts. However, this stage introduces challenges such as distortion and anisotropic shrinkage, which directly affect dimensional accuracy. Many studies have explored general sintering behavior and sintering shrinkage, but the current literature lacks a comprehensive understanding of anisotropic sintering shrinkage in binder-jet-fabricated stainless steels. Specifically, the coupled effects of sintering kinetics, sintering temperature, temperature-dependent viscosity, porosity distribution, activation energy, alloy composition, and microstructural evolution on anisotropic shrinkage in 316L and 17-4 PH have not been investigated. This gap limits predictive capability and effective process optimization for dimensional control in BJ components.
2. Powder size distribution (PSD) is a key factor governing sintering kinetics, green part density, and the overall quality of binder jet additively manufactured (BJAM) components. For 316L, several works have successfully tailored BJ printing and sintering parameters to achieve optimum final densities. However, a clear understanding of how particle size distribution alone affects anisotropic shrinkage, densification, and microstructural evolution in binder-jetted 316L stainless steel is still lacking.
3. The final BJ part quality and crashworthiness are important for the applicability of stainless steels in the automotive industry. So far, most researchers have focused on the effect of BJ printing parameters on quasi-static mechanical properties. However, there is no existing work on the dynamic compressive response of binder-jetted 316L stainless steel under high strain-rate conditions. In particular, the combined effects of strain rate, temperature, powder size distribution, and HIP treatment on the high-strain-rate mechanical behavior and underlying deformation mechanisms of stainless steels have not been studied.

4.2 Research Objectives

This project aims to investigate the process-microstructure-property relationship for binder jetting in two different stainless steels – high-density austenitic (316 L) and martensitic (17-4 PH). Based on the previously identified gaps, the specific objectives are defined as follows:

- 1. Investigate the anisotropic sintering behavior for BJ stainless steels of 316L and 17-4 PH.**
 - a) Measure in-situ directional sintering shrinkage along the X-, Y-, and Z-axes using dilatometry across a broad sintering temperature range.
 - b) Determine the dominant sintering mechanisms by extracting activation energies and identifying the transition from grain-boundary to volume diffusion.
 - c) Characterize microstructural evolution, including porosity morphology, grain growth, and phase transformations (martensite, retained austenite, and δ -ferrite) as a function of temperature and printing axis.
 - d) Develop and calibrate a physically-based viscoplastic sintering finite-element model to capture direction-dependent shrinkage, viscous moduli, and pore-driven anisotropic deformation during sintering.
- 2. Investigate the effect of powder size distribution (PSD) on anisotropic sintering behavior in BJ 316L stainless steel.**
 - a) Quantify directional sintering shrinkage (X and Z directions) as a function of temperature using dilatometry.
 - b) Compare densification kinetics and final relative density between MIM-cut (narrow PSD) and LPBF-cut (broad PSD) powders.
 - c) Analyze the evolution of microstructure, including porosity, grain size, and δ -ferrite formation, to identify mechanisms governing anisotropic shrinkage.
 - d) Evaluate the anisotropic shrinkage ratio (ASR) to establish the relationship between PSD, shrinkage magnitude, and sintering anisotropy.
- 3. Study the high-strain-rate compressive behavior of binder-jetted 316L stainless steel.**

- a)** Characterize the strain-rate- and temperature-dependent compressive response of binder-jetted 316L stainless steel using Split Hopkinson Pressure Bar (SHPB) testing over strain rates of $500\text{--}2000\text{ s}^{-1}$ and temperatures from room temperature to $600\text{ }^{\circ}\text{C}$.
- b)** Quantify the effects of powder size distribution (MIM-cut vs. LPBF-cut) on dynamic strength, strain-rate sensitivity, and deformation stability under high-strain-rate loading.
- c)** Evaluate the influence of hot isostatic pressing (HIP) on high-strain-rate mechanical behavior by comparing as-sintered and HIP-treated conditions.
- d)** Correlate dynamic mechanical response with microstructural features, including grain size, porosity, δ -ferrite content, subgrain structure, and deformation-induced lattice misorientation using SEM and EBSD analysis.

CHAPTER 5 ARTICLE 1: Anisotropic sintering behavior of stainless steel 316L printed by binder jetting additive manufacturing

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Abstract

The influence of sintering temperature and printing orientation on the anisotropic sintering shrinkage of binder jetted (BJ) stainless steel 316L (SS316L) was investigated. Near quasi-isotropic shrinkage behavior with very little anisotropy ($\leq 4\%$) in the powder bed plane was observed. The out-of-plane anisotropic shrinkage of up to 16% was attributed to the primitive lines formed perpendicular to the build direction. This follows the power law, and the activation energy is unaffected by anisotropy. For all printing orientations, grain boundary diffusion (25-162 kJ/mol) was the dominant sintering mechanism between 1100 °C and 1300 °C. Above 1300 °C, volume diffusion (490-623 kJ/mol) takes over as the dominating sintering mechanism, leading to significantly higher shrinkage up to 15%. The anisotropic shrinkage behavior does not affect the microstructure, as a similar evolution of grain sizes, ferrite fractions, and bulk densities (up to 96%) is observed upon sintering.

Keywords: Additive manufacturing, Binder jetting, Anisotropy, 316L, Sintering

5.1 Introduction

Additive manufacturing (AM) is a rapidly evolving technology offering the possibility to construct three-dimensional (3D) parts with complex geometries [1,2]. A wide assortment of metals, such as nickel [118–122], aluminum [123–125], magnesium [126,127], and stainless steel [128–130], can be processed using AM. However, most AM processes involve melting and rapid solidification, which can promote the formation of brittle phases in materials with complex alloy chemistries

[131]. This results in in-process defects such as cracking [132,133] and residual stresses [134,135], which can adversely affect the final part quality.

Binder jetting (BJ) is a high-throughput, cost-effective AM process appealing for high-volume part production in the medical and automotive industries [3,4,25,34]. Unlike laser AM, BJ utilizes inkjet printing technologies to bond particles layer-by-layer at room temperature. This suppresses the formation of in-process defects such as solidification cracking, residual stresses, and distortions attributed to wide solidification windows and large thermal gradients [136]. Printed (green) parts need to undergo a sintering heat treatment to produce the final near-net shape components [3,4,137]. During sintering, green parts experience shrinkage, densification, and strengthening as particles fuse together under the influence of heat. This introduces several challenges, such as material deformation and anisotropic sintering shrinkage, limiting the final part's dimensional accuracy [138].

Anisotropic sintering shrinkage, particularly, can reduce the final part dimensions by up to 20% [139]. This occurs due to uneven reduction in part size during the sintering process. Irregularities such as primitive lines, porosity variation, and binder distribution inconsistencies inherent in the green parts promote anisotropic sintering shrinkage [140]. These irregularities primarily arise from the operational mechanics of the printer [141]. For example, powder PSD, powder shape, powder spreading, printhead motion, jetting, saturation, and build plate adjustment can introduce directional irregularities, which can affect the density and binding strength across different directions in the green part [139,142,143].

Binder jetting stainless steel (SS) is of particular interest due to its remarkable corrosion resistance, strength, and durability, making it ideal for advanced engineering applications [55]. To date, few papers [139,144] have investigated the anisotropic sintering shrinkage of SS316L (316L). Ziaee et al. [145] reported significant sintering shrinkage for BJ 316L in all three axes with respect to the powder bed at only one sintering temperature of 1360 °C. The authors also reported the highest dimensional reduction in the build direction, attributed to more porosity between layers. Olevsky et al. [146] and Zago et al. [147] used sintering models for 316L to show anisotropic deformation due to non-uniform gravitational effects within the part. Recently, Rios et al. [139] investigated the anisotropic sintering behavior of BJ 316L using dilatometry, reporting significant shrinkage along the build direction during sintering. All presented works focused on high-temperature (>1300°C)

anisotropic behavior and fine unimodal powder ($D_{90} \leq 20 \text{ }\mu\text{m}$). Moreover, the effect of microstructure and activation energy for sintering on the anisotropic behavior was not investigated.

An in-depth study of the anisotropy sintering phenomenon needs to be conducted to improve the shrinkage simulation accuracy and optimize the dimensional accuracy of a BJ 316L sintered part. This study aims to first quantify the anisotropic sintering phenomenon not only along the build direction but also within the powder bed plane for BJ 316L. A wide range of sintering temperatures (1100-1390 °C) is considered for powder with bimodal distribution as the feedstock material. Previously, Batmaz et al. [129] showed better sintering capability of a 316L powder distribution ($D_{90} \leq 40 \text{ }\mu\text{m}$) due to a higher initial green density. The in-plane and build direction anisotropy behavior is correlated to microstructure features such as pores, grains, and phases to better understand the underlying mechanism associated with the anisotropic behavior during sintering of BJ 316L.

5.2 Experimental procedures

5.2.1 Materials

Stainless steel 316 L powder produced by gas atomization was used in this study. The powder is mainly spherical with a few satellite particles attached to larger powder particles, as shown in Fig. 1. The particle size distribution (PSD), including $D_{10}=9 \text{ }\mu\text{m}$, $D_{50}=20 \text{ }\mu\text{m}$, and $D_{90}=40 \text{ }\mu\text{m}$, was obtained using the CAMSIZER X2 system (RETSCH, Wuppertal, Germany). The nominal chemical composition is provided in Table 1.

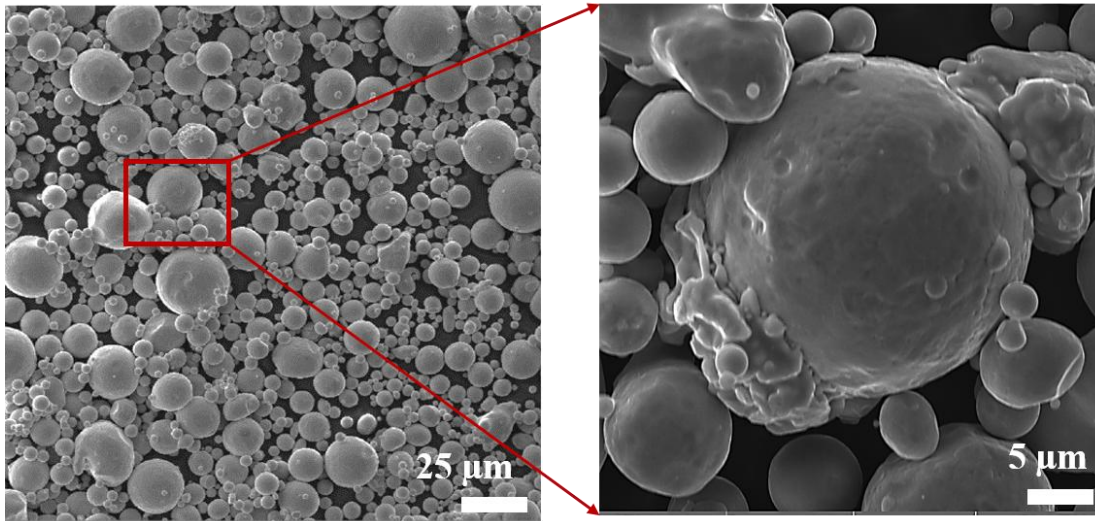


Figure 5-1 SEM micrograph of gas-atomized SS 316L powder.

Table 5.1. Powder compositions of SS 316L in wt.%.

Fe	Cr	Ni	Mo	Mn	Si	N	O	P	C	S
Bal.	16.5-18	10-13	2-2.5	2.00	1.00	0.13	0.06	0.04	0.03	0.03

5.2.2 Binder jetting

A GE Additive series 2 binder jet system was used to print 316L powders. BJ samples were produced with a GE proprietary water-based binder. Cubic samples with a cross-sectional area of $10 \times 10 \text{ mm}^2$ and 20 mm length were printed in three orientations, the X-axis, Z-axis, and Y-axis, as shown in Fig. 2. The X-axis shows the printhead movement and the recoater direction, the Z-axis shows the build direction (BD), and the Y-axis is normal to X and Z axis. The processing parameters are provided in Table 2 and the printed samples are called green parts herein. Green densities were determined using the geometrical approach by measuring the volume and weight of each green part.

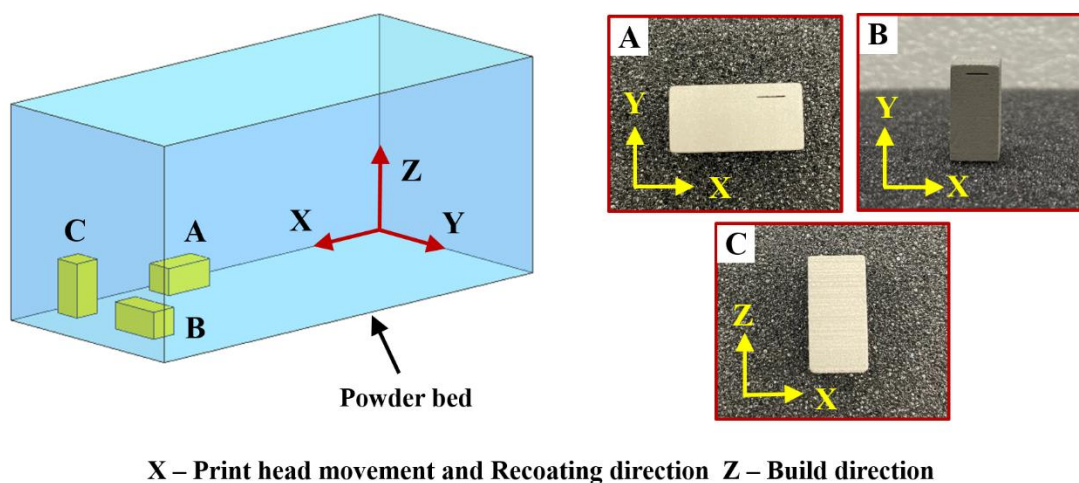


Figure 5-2 Schematic showing setup for binder-jet samples A, B, and C printed in three different orientations, X-axis, Y-axis, and Z-axis, concerning the powder bed. The X-axis shows the printhead movement and the recoating direction, Y axis is normal to X and Z axis and the Z-axis shows the build direction. Images of the printed (green) samples are shown to the right of the schematic. All samples have a cross-sectional area of $10 \times 10 \text{ mm}^2$ and are 20 mm long.

Table 5.2 Binder jet printing parameters for stainless steel 316L.

Build parameter	Value
Binder saturation (%)	100
Binder set time (s)	10
Bed temperature ($^{\circ}\text{C}$)	65
Recoating speed (mm/s)	90
Layer thickness (μm)	100

5.2.3 Dilatometry

A thermal gravimetric analysis (TGA) test was carried out to determine the optimal debinding conditions for the binder system. Debinding was performed using heat treatments conducted in a pure H_2 atmosphere. The green parts were heated from room temperature at a rate of $5^{\circ}\text{C}/\text{min}$ up to 190°C , held there for 60 minutes, then green parts are debounded between $280\text{--}620^{\circ}\text{C}$ for total

time of 5.5 hours, and finally heated to 890°C at 2°C/min, with a 5 hours hold to facilitate binder decomposition and get some presintered strength before moving the samples to sintering step. Debound samples were placed in a Netzsch DIL 402S dilatometer to perform dilatometry sintering. The longitudinal axis of each sample was aligned parallel to the pushrod with a contact force of 2.94 mN to measure dimensional changes and sintering shrinkages in the X-, Y-, and Z-axes. The other faces of the samples were free from friction during the sintering experiments. Sintering was performed in an Argon-5%H₂ atmosphere flowing at 100 mLmin⁻¹. A schematic of the heat treatment profile for dilatometry sintering is shown in Fig. 3a. The sintering treatment consisted of three stages, heating, hold, and cooling. The samples were heated at 10 °C/min from room temperature up to the sintering temperature (red-dashed line), held for 6 hr dwell time, and cooled back to room temperature at 10 °C/min. This was repeated for six different sintering temperatures for the X-, Y-, and Z-axes, as shown in Fig. 3b. The sintered density was measured using the Archimedes method. Two dilatometry sintering tests were performed for each sintering condition and printing orientation to account for experimental variability and repeatability, as shown in Fig. 3b.

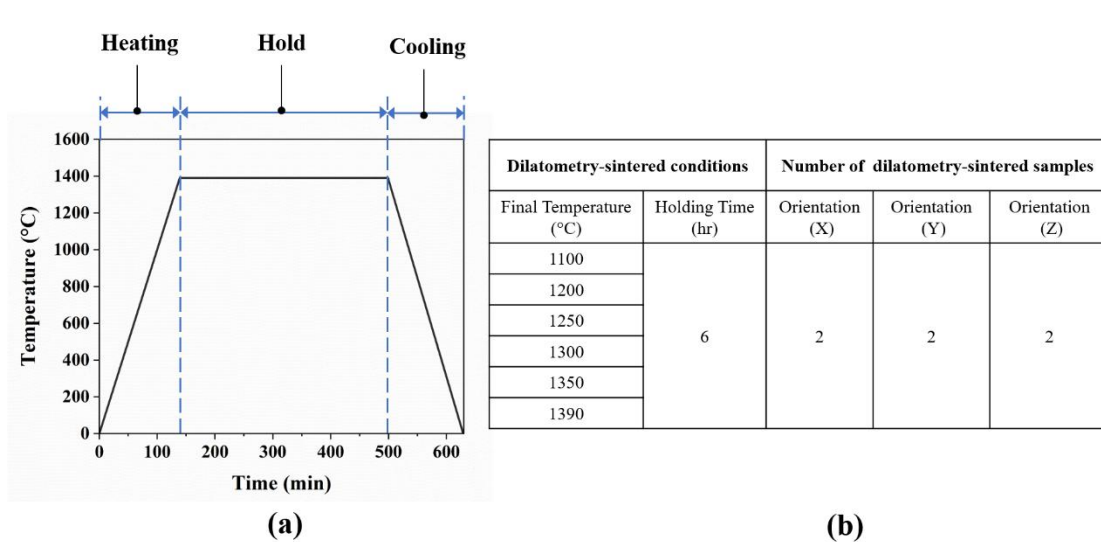


Figure 5-3 Dilatometry sintering of BJ SS 316L. (a) The heating cycle includes heating at 10 °C/min from room temperature up to a target sintering temperature, following hold for 360 min, and cooling at 10 °C/min. (b) Sintering conditions include sintering temperature, dwell time, and number of repetitions.

5.2.4 Microstructure characterization

Microscopic analyses were conducted to examine the microstructure before and after sintering. Sintered samples were sectioned along the printing orientation to study the effect of printing orientation and sintering temperature on microstructure. Sample preparation involved conventional grinding using abrasive paper and polishing with diamond suspension, followed by a 0.04 μm colloidal silica final finish. The polished samples were ultrasonically cleaned in distilled water for 5 min. Optical microscopy was performed using a Keyence VHX7000 optical microscope to observe pores and perform grain size quantification of both green and as-sintered parts. The line intercept method, per ASTM E112–13 [148] for three images, was used to determine the average grain size using the optical micrographs. A JEOL JSM-7600F field emission gun scanning electron microscope (FEG-SEM) equipped with an Oxford Instruments X-Max 80 mm energy dispersive spectroscopy (EDS) detector was used to examine the microstructure and phase characteristics for different sintering conditions.

X-ray diffraction (XRD) was conducted using a Bruker D8 Advance diffractometer with $\text{Cu K}\alpha$ radiation to identify and quantify the volume fraction evolution of delta ferrite. The diffraction instrument was operated at 40 kV and 40 mA with a 40–90° 2θ range and 0.04° step size. Phase identification was carried out using DIFFRAC.EVA software with the ICDD-PDF4+ database. The Rietveld method was integrated into the software to measure the volume fraction of the secondary phases.

5.3 Results

5.3.1 Green density

All printed samples produced similar green densities (58-60%) and different pore distributions varying with printing orientation. Fig. 4 shows the surfaces of as-printed 316L (green state) in the X-, Y-, and Z-axes. For all cases, pores typical of green parts are observed on the sample surface. For the X- and Y-axes, pores are randomly distributed, while for the Z-axis, they form periodically along the build direction and are aligned along primitive lines perpendicular to the build direction. This is inherent to the BJ process and is commonly observed by other researchers [149,150].

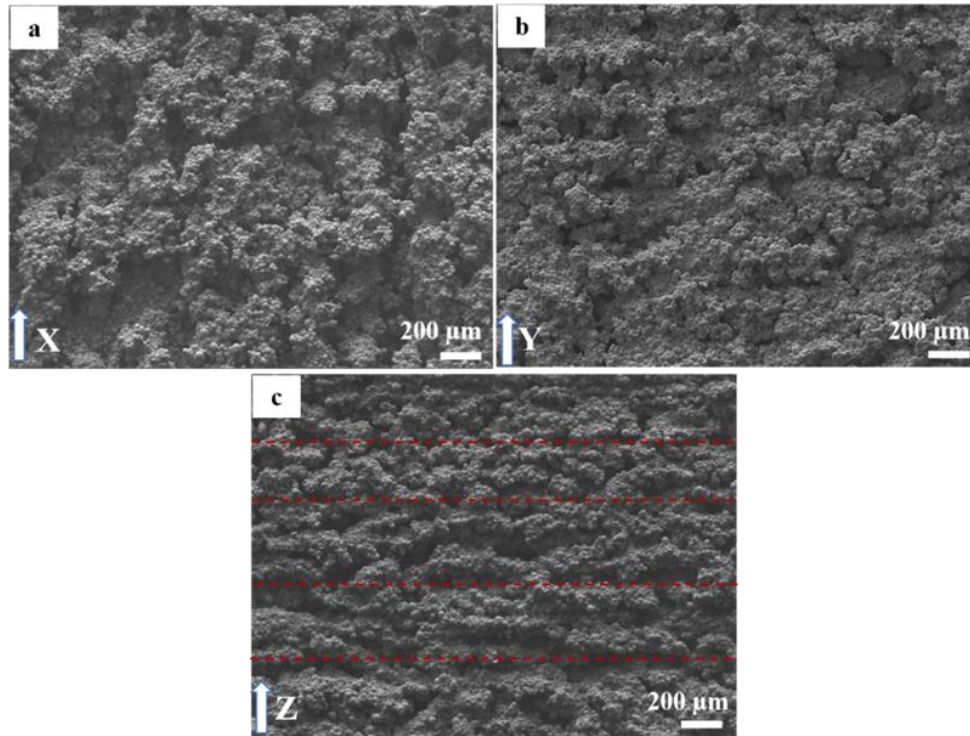


Figure 5-4 SEM micrographs of green samples in the (a) X-axis, (b) Y-axis, and (c) Z-axis. The red dashed lines in (c) indicate primitive lines perpendicular to the build (Z) direction.

5.3.2 Dilatometry Sintering

Sintering stages

To explain the sintering stages, a sample heating profile and dilatometry curve of BJ 316L sintered at 1390 °C in the Z-axis are plotted in Fig. 5. The heating profile (blue) is taken from Fig. 5, where t_s and t_f define the start and end points of dwell time. The dilatometry curve (black) shows the shrinkage percentage (Y-axis) in response to time (X-axis). Initially, upon heating to the onset sintering temperature (960 °C), the material expands by ~1% from 0 to t_0 (onset sintering time). As the temperature increases, the material shrinks, and the shrinkage rate varies with time from t_0 to t_f . Three different slopes, m_1 (pink-dashed line), m_2 (cyan-dashed line), and m_3 (orange-dashed line), are measured within this region to distinguish changes in the sintering shrinkage rate. During the heating cycle, between t_0 and t_s , there is a small window with rapid changes in the sintering rate characterized by m_1 and m_2 . During the isothermal section of the heating profile, between t_s and t_f , the shrinkage rate is defined by m_3 .

Fig. 6 shows the amount of shrinkage in different printing orientations at different sintering temperatures. The shrinkage values in the X-axis (Fig. 6a) and Y-axis (Fig. 6b) are similar; however, the shrinkage values in the Z-axis (Fig. 6c) are higher. For example, at 1350 °C, samples shrink by 12.00% in the X-axis and 12.53% in the Y-axis compared to 14.22% shrinkage in the Z-axis. For all printing orientations, shrinkage increases with increasing temperature. For example, in the X-axis, shrinkage increases from 5.67% at 1100 °C to 12.51% at 1390 °C. Shrinkage in the Z-axis becomes more pronounced with increasing temperature. As the temperature increases from 1100 °C to 1390 °C, shrinkage increases from 5.91% to 15.02%.

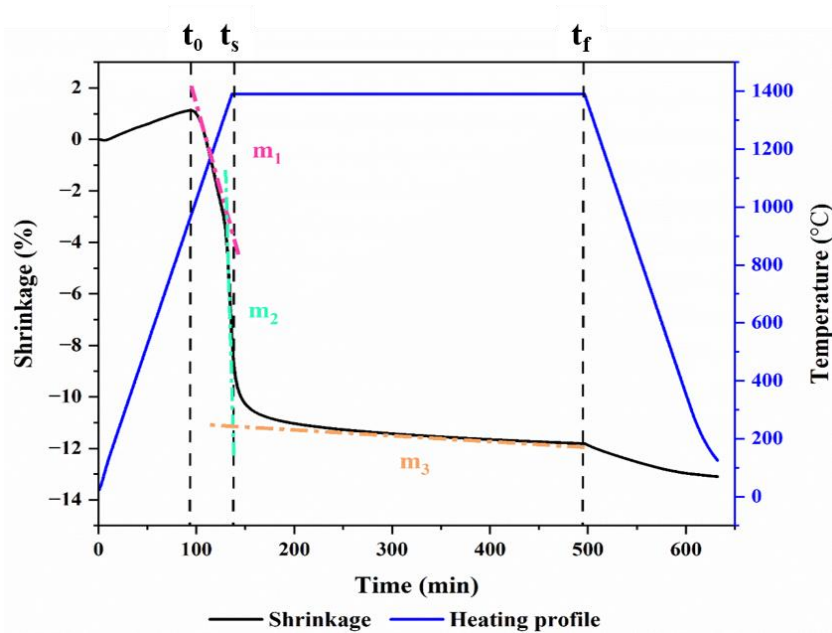


Figure 5-5 Shrinkage vs. time for different shrinkage regimes, m_1 , m_2 , and m_3 , observed during dilatometry sintering of BJ SS316L. The sample sintered at 1390 °C in the Z-axis distinguishes the regimes.

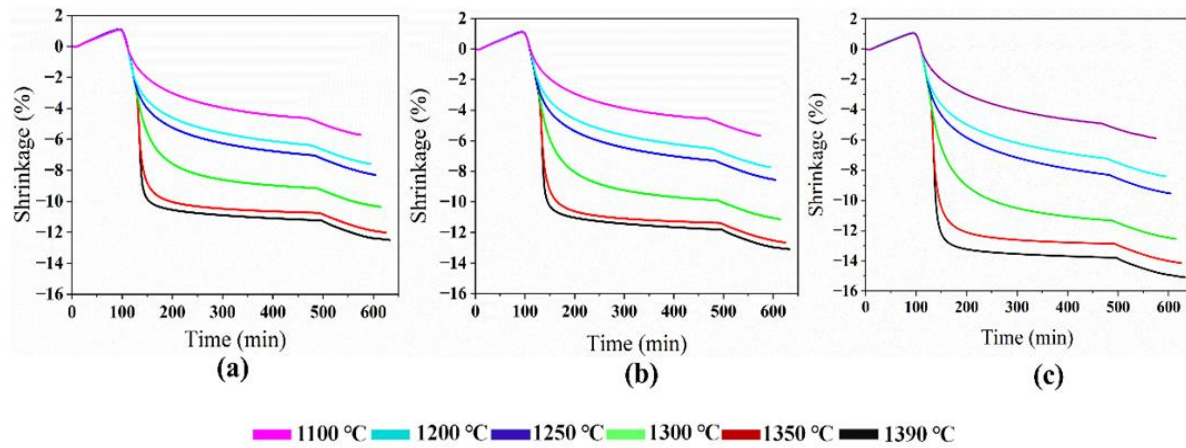


Figure 5-6 The effect of temperature on shrinkage percentage during sintering for (a) X-axis, (b) Y-axis, and (c) Z-axis with respect to the powder bed.

5.3.3 Effect of sintering temperature on microstructure and phases

Fig. 7 displays SEM micrographs of BJ samples sintered at various temperatures for three printing orientations: X-axis (Fig. 7a), Y-axis (Fig. 7b), and Z-axis (Fig. 7c). Each micrograph includes information on the sintered density of the respective sample and the vertical axis is aligned along the printing orientation. These images provide valuable insights into the solid volume fraction and pore distribution behavior for different sintering conditions. For all printing orientations, the solid volume fraction increases as the temperature increases from bottom to top, which explains higher sintered density up to ~96% at 1390 °C. Pore distribution shifts from irregular, interconnected pores to more isolated, equiaxed pores with increasing temperature. Interconnected pore alignment is apparent along preferential orientations from 1100 °C up to 1300 °C for all printing orientations. In the X- and Y-axes, pores are aligned along the printing orientation (vertical, red-dashed lines in Fig. 7a, 7b), whereas in the Z-axis, pores are aligned perpendicular to the build direction (horizontal, red-dashed lines in Fig. 7c). During the BJ process, when the high-velocity binder droplet interacts with the powder bed, particles are ejected due to the transfer of momentum from the binder droplet to the powder particles, resulting in the creation of primitive lines [24,149]. In this study, such primitive lines produce significantly higher shrinkage (up to 15%) for samples oriented in the Z-axis, as shown in Fig. 6c.

To better assess the underlying metallurgical factors affecting sintering shrinkage, high magnification SEM images are examined in Fig. 8 for the X-axis (Fig. 8a), Y-axis (Fig. 8b), and

Z-axis (Fig. 8c) printing orientations. For all printing orientations, the microstructure is similarly sensitive to sintering temperature. At lower sintering temperatures (1100 °C, 1200 °C), a high porosity level with open and irregularly shaped pores is observed, and the microstructure mainly consists of the austenite (parent) phase. Irregularly shaped pores result from incomplete consolidation and limited particle bonding [151]. As the temperature increases to 1300 °C, pores become localized at triple-grain boundary junctions, and a secondary phase (red arrows) becomes apparent. Above 1350 °C, most pores become tiny and isolated within the grains, and the secondary phase fraction increases. Twin boundaries (gold arrows) observed in Fig. 8 represent annealing twins, typical of the 316L microstructure [87].

The phases observed above 1300 °C are characterized using EDS elemental mapping of a sample sintered at 1390 °C in Fig. 9. The grain boundaries are decorated with two distinct phases rich in Mo and Cr. One phase is ~20 µm large and mainly depleted in Ni, typical of the delta ferrite (δ -ferrite) phase in 316 L [150]. Another phase is ~2 µm thin and is depleted in Ni, Fe, and Mn, typical of the sigma (σ) phase in 316 L [152].

Fig. 10 shows the XRD phase analyses of specimens sintered at different temperatures in the Z-axis (Fig. 10 (a)) and the X-axis (Fig. 10 (b)). For both printing orientations, austenite dominates over a small fraction of the δ -ferrite phase in the sintered microstructure. Ferrite formation is dependent on the sintering temperature. At lower temperatures (<1300 °C), no δ -ferrite peak is observed in Fig. 10 (a) and (b). As the temperature exceeds 1300 °C, the δ -ferrite peak becomes apparent with up to ~8% detected at 1390 °C in Fig. 10 (c) for the two samples orientations. This is consistent with previous observations of γ - δ phase transformation around 1300°C [150,153].

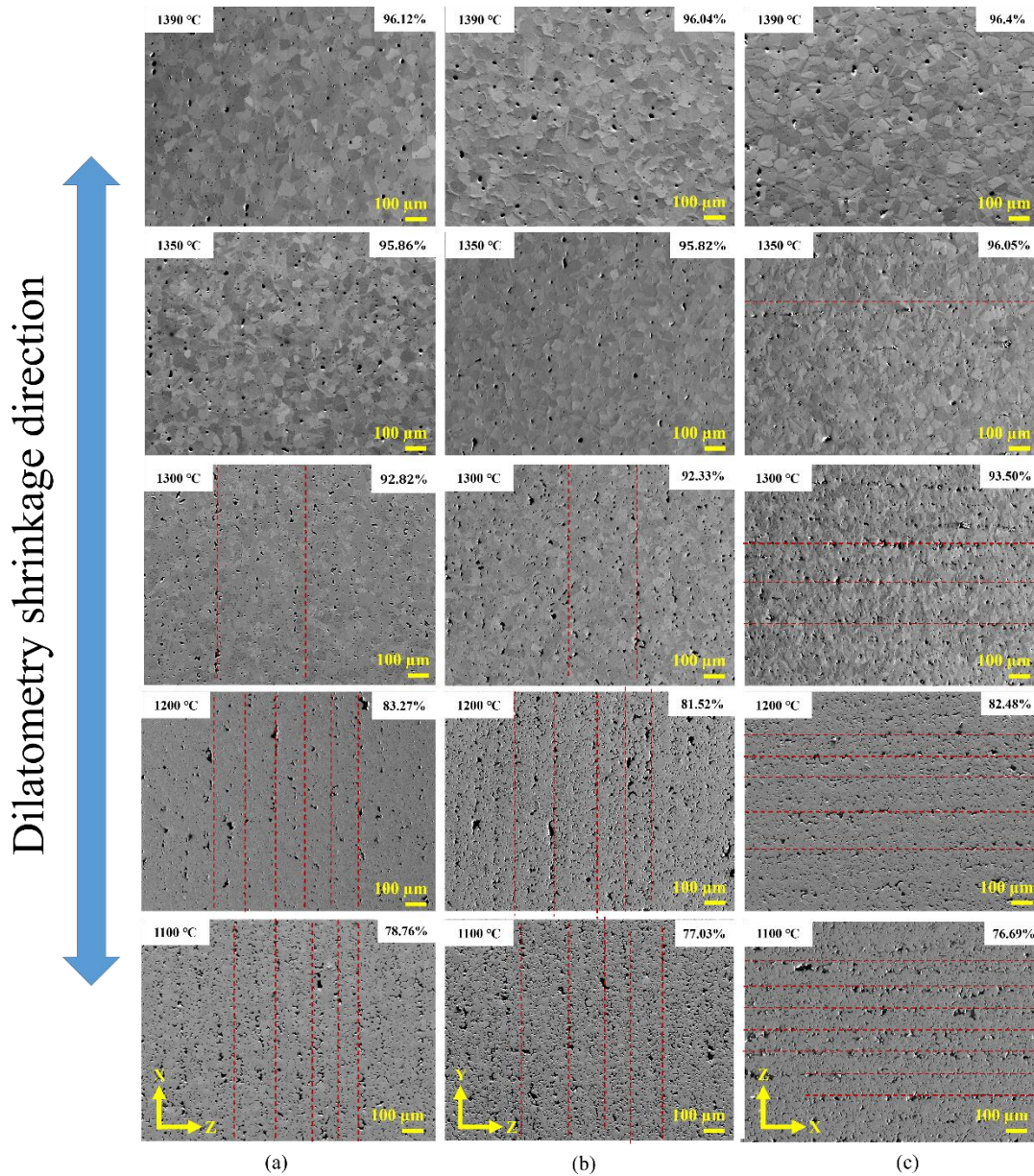


Figure 5-7 SEM micrographs of sintered samples with a holding time of 6 hr at various sintering temperatures at (a) X-axis, (b) Y-axis, and (c) Z-axis printing orientations. Red-dashed lines indicate pore alignment, and the vertical axis of each micrograph represents the sample printing orientation.

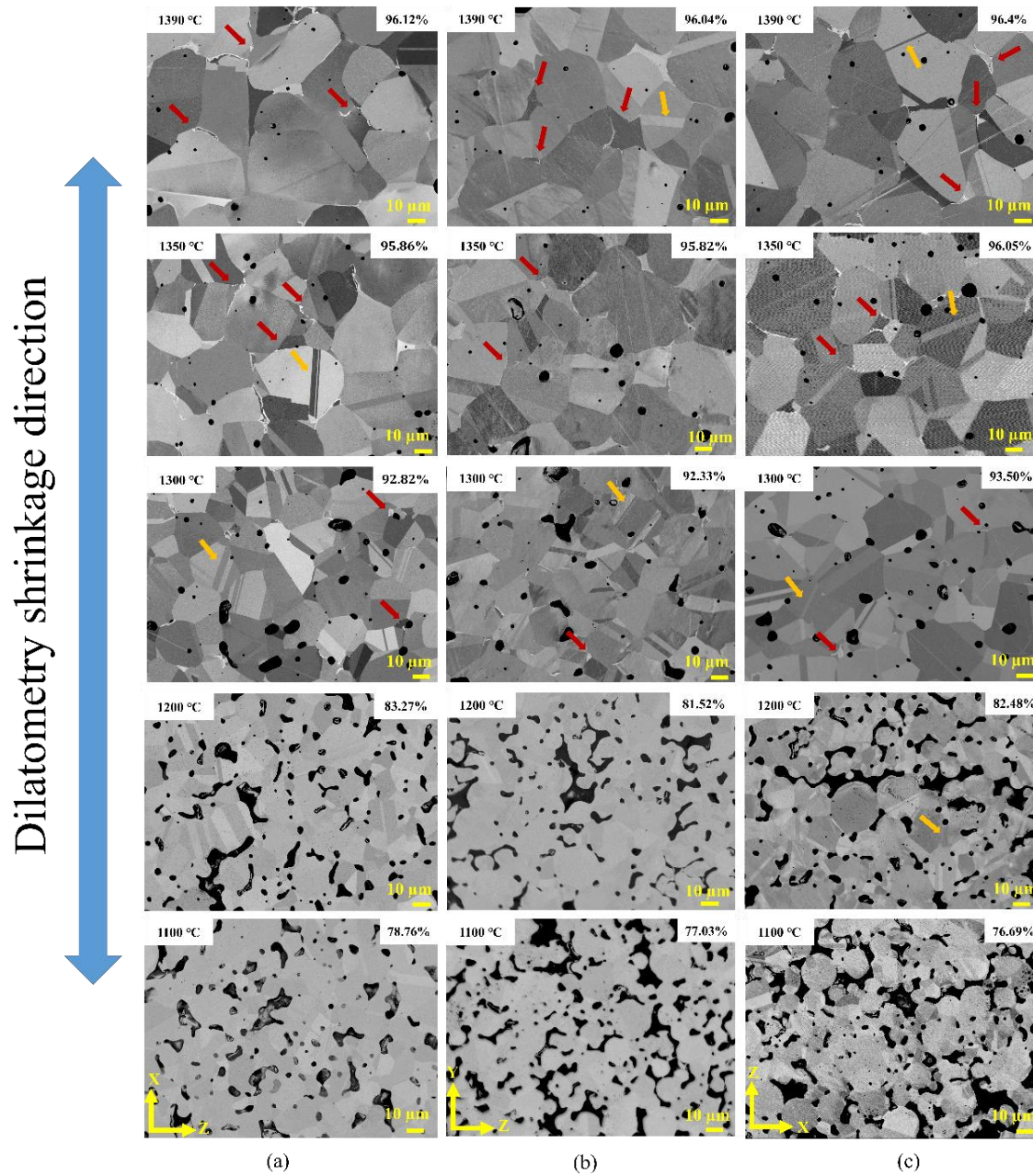


Figure 5-8 SEM images of BJ SS 316L sintered at five different temperatures between 1100 °C and 1390 °C for the (a) X-axis, (b) Y-axis, and (c) Z-axis printing orientations. Secondary phases are highlighted with red arrows, and twin boundaries are highlighted with gold arrows. The vertical axis of each micrograph represents the printing orientation.

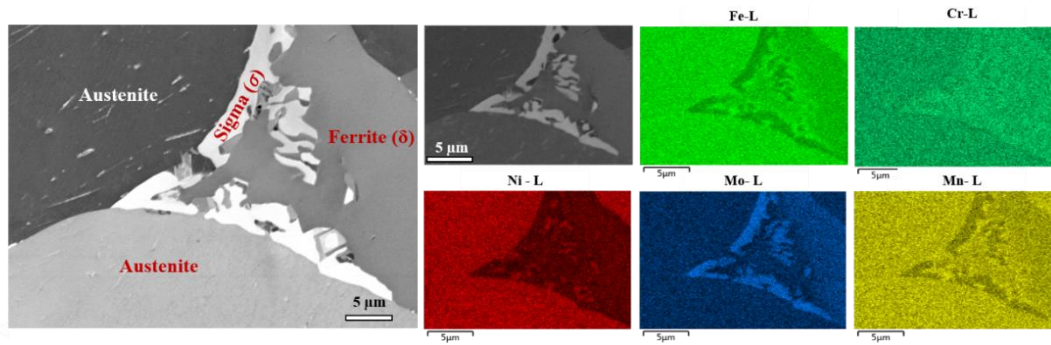


Figure 5-9 SEM micrograph and corresponding EDS map showing elemental distribution at the sintered microstructure of BJ SS 316L in 1390 °C.

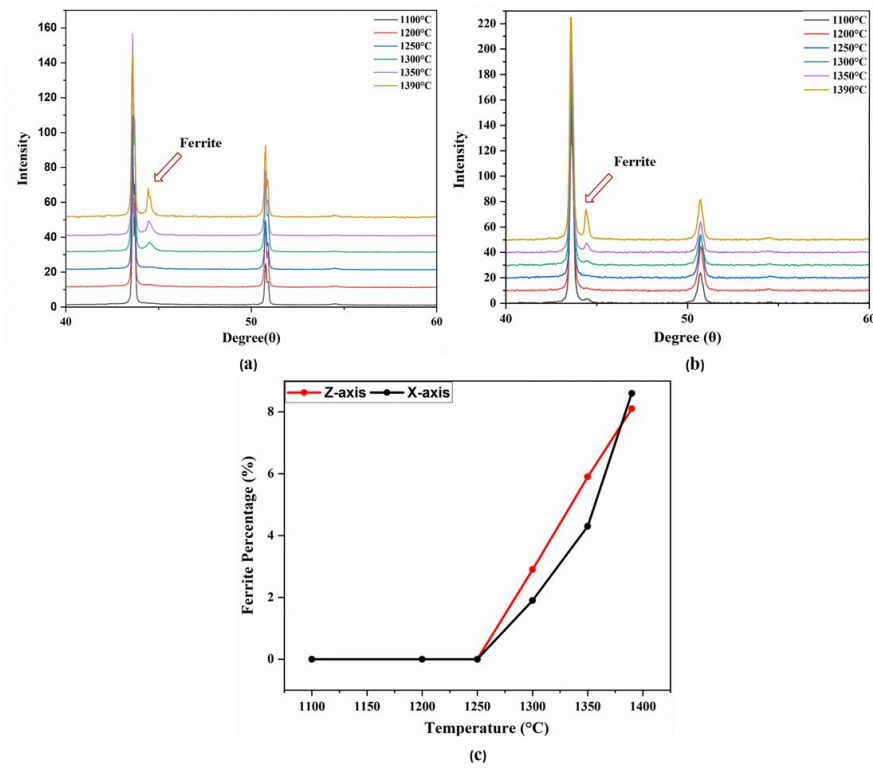


Figure 5-10 XRD patterns of BJ SS 316L sintered at different temperatures in the a) Z-axis and b) X-axis relative to the powder bed. (c) Effect of sintering temperature on ferrite percentage for X- and Z-axes.

5.4 Discussion

5.4.1 Effect of sintering temperature on shrinkage

Three distinct shrinkage regimes (m_1 , m_2 , and m_3) were identified during the sintering of BJ 316L parts in Fig. 5. The appearance of shrinkage regimes mainly depends on the sintering temperature, as shown in Fig. 6. The m_1 regime dominates shrinkage in dilatometer curves at low temperatures (1100-1300 °C) for all printing orientations. The sintering rate m_1 increases linearly until 1300 °C then remains relatively constant between -0.16 and -0.18 %/min in Fig. 11a. Above 1300 °C, the sintering rate m_2 becomes dominant with values ~ 7 times (7X) greater than m_1 , ranging between -0.7 and -0.9 %/min.

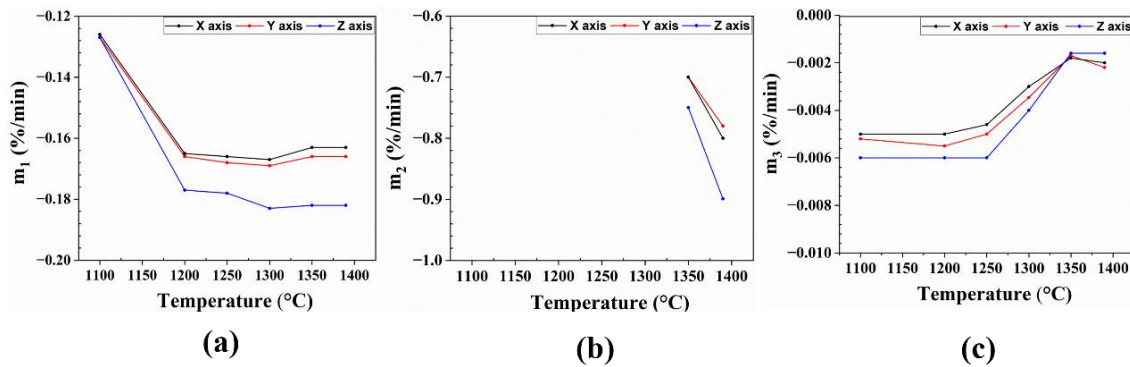


Figure 5-11 The sintering rate associated with the (a) m_1 , (b) m_2 , and (c) m_3 regimes during the sintering cycle in Fig. 6 for printing orientations X-, Y-, and Z-axes.

The m_3 regime, corresponding to the isothermal section of the sintering cycles, is shown in Fig. 11 (c). Initially, the sintering rate is relatively low (-0.005-0.006 %/min) and remains constant for all printing orientations up to 1250 °C. As the sintering temperature increases further, the sintering rate decreases to -0.002 %/min up to 1350 °C, above which the value becomes constant. This means that most shrinkage occurs during the heating cycle (Fig. 6) when the m_2 regime becomes dominant above 1250 °C. This is because (i) the shrinkage rate in m_2 is significantly higher than in m_1 , and (ii) there is more time for shrinkage to occur as the target temperature increases.

5.4.2 Activation energy for sintering of binder jet 316L

The change in shrinkage rate from m_1 to m_2 suggests a change in the sintering mechanism with temperature. The active mechanisms in these regimes can be determined using the activation energy for material densification, based on Johnson et al. [154], using equation (2):

$$\ln\left(-T \frac{dL}{dtL_0}\right) = -\frac{Q}{RT} + \ln\left(\frac{F(\rho)AD_0}{G^c}\right) = -n\left(\frac{1}{T}\right) + K, \quad (2)$$

where dL/L_0 is the time-dependent linear shrinkage obtained by dilatometric testing as a function of temperature (T), Q is the activation energy, R (8.314 J/mol•K) is the gas constant, $F(\rho)$ is a function of density, A and c are material constants, D_0 is the initial diffusion coefficient, and G is the grain size.

To explain the active sintering mechanisms at different temperatures, $\left(\ln\left(-T \frac{dL}{dtL_0}\right)\right)$ versus $1/T$ is plotted in Fig. 12 using the 1390 °C dilatometer results obtained from Fig. 5. The slope (n) is used to determine the activation energy (Q) based on $-Q/R$ from equation (2) [151,155–158]. The measured slopes and calculated Q values, corresponding to the temperature-dependent regimes, m_1 and m_2 , observed in Fig. 5a are shown in Fig. 12. Note that similar activation energies were obtained for all dilatometer curves taken at different temperatures in Fig. 6 (c) (not shown here).

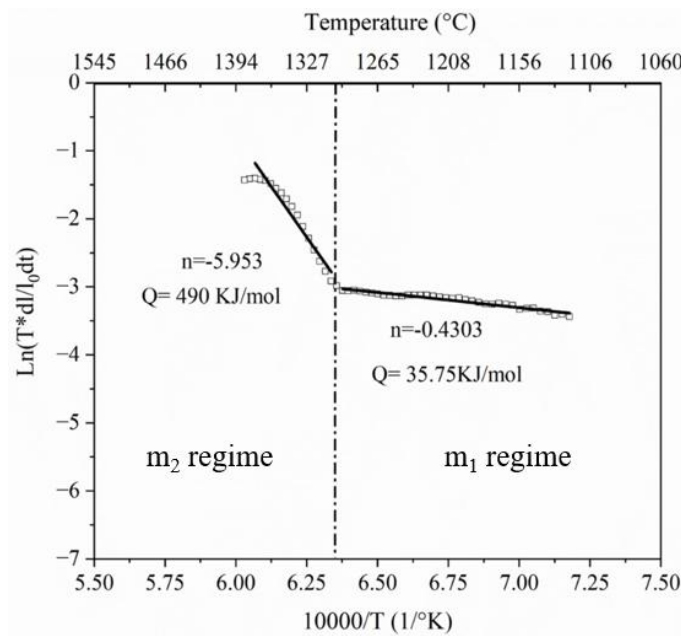


Figure 5-12 The effect of temperature on activation energy (Q) in different sintering shrinkage regimes for the sample printed in the Z-axis and sintered up to 1390°C. The slopes (n) correspond to the m_1 observation range (1100-1300°C) and m_2 observation range (1300-1390°C). The two regimes are separated by the vertical dash line.

At lower target sintering temperatures (1100-1300 °C) in Fig.6, shrinkage was controlled by the m_1 regime, corresponding to low activation energies (25-162 kJ/mol). Low activation energies

below 200 kJ/mol suggest the grain boundary diffusion mechanism [159]. Grain boundary diffusion occurs when atoms migrate along the interface between individual grains in the material [151]. The effect of sintering temperature on the grain size and relative density of sintered BJ 316L here is presented in Fig. 13. When the sintering temperature is low (≤ 1300 °C), the grain size remains similar (23-32 μm) in Fig. 13a while the relative density increases (76-94%) in Fig. 13b with increasing temperature. This suggests that the relative density is not affected by grain boundary mobility and is mainly driven by pore coalescence at the GBs due to GB diffusion. Smaller grain sizes at lower sintering temperatures correspond to pores situated along GBs (see Fig. 8c). Pores act as pinning sites, exerting Zener drag on the moving grain boundaries and inhibiting grain growth [160]. This limits material densification, resulting in small shrinkage ($\sim 4\%$ in Fig. 6) and low sintered densities ($< 94\%$ in Fig. 13b) below 1300 °C.

At higher target sintering temperatures (> 1300 °C) in Fig. 6, the shrinkage is dictated by the m_2 regime, associated with higher activation energies (490-623 kJ/mol), as shown in Fig. 12. This corresponds to mass transfer and material densification which mainly occur via volume diffusion [159–161]. Volume diffusion involves the movement of atoms through the material's crystal lattice. At elevated temperatures, atoms have increased kinetic energy, allowing them to diffuse more rapidly through the lattice. Within the bulk of the grains, there are fewer barriers to atomic motion (GB energy, impurities segregated at the GBs), resulting in a more uniform distribution of the pores after sintering [61,162]. This is observed in Fig. 8, where pores can now be observed in the center of grains. This reduces the Zener dragging forces, allowing more GB migration to occur. This results in larger grain sizes (37-56 μm in Fig. 13a), increased sintering shrinkage ($\sim 15\%$ in Fig. 6c), and higher final densities ($\sim 96\%$ in Fig. 13b).

The increase in shrinkage between 1300 °C and 1390 °C also coincides with higher ferrite fraction (3-8%) compared to lower temperatures observed in Fig. 10c. Ferrite formation is attributed to a higher rate of self-diffusion in the body-centered cubic (BCC) compared to the face-centered cubic (FCC) structure of austenite [163]. Faster self-diffusion allows the particles to bond quickly during sintering, resulting in faster consolidation. Interphase boundaries such as (γ - δ) are more effective than γ grain boundaries in trapping the porosity and improving densification [87,163]. Hence, volume diffusion ($Q > 500$ kJ/mol) at higher sintering temperatures (> 1300 °C) dominates over GB diffusion ($Q < 200$ kJ/mol) at lower temperatures (≤ 1300 °C).

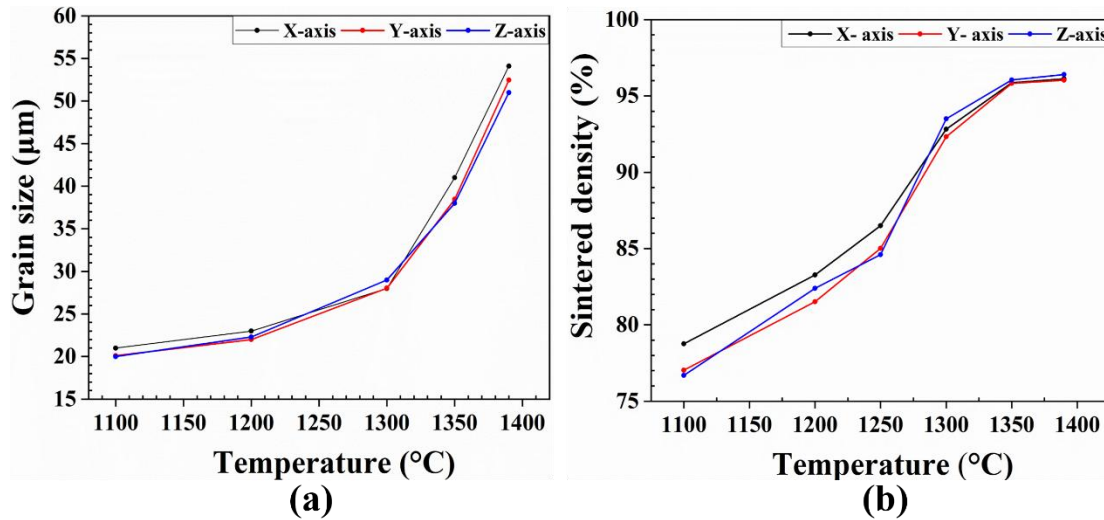


Figure 5-13 Effect of sintering temperature on the (a) grain size and (b) final density of sintered BJ SS 316L samples printed in the X-, Y-, and Z-axes.

5.4.3 Anisotropic shrinkage

The shrinkage is noticeably different between the three printing orientations in Fig. 6. Fig. 14 (a) compares the maximum strains as a function of temperature for the X-, Y-, and Z-axes. The maximum shrinkage increases linearly for all printing orientations, followed by a sharp increase above 1250 $^{\circ}\text{C}$. This increase is caused by a transition from lower to higher activation energy at 1300 $^{\circ}\text{C}$ facilitating sintering (see section 4.1). For all temperatures, shrinkage is more important along the build direction when compared to the X-axis and Y-axis. This is because primitive lines along the build direction generate more shrinkage. Above 1300 $^{\circ}\text{C}$, the decreased change in shrinkage rate coincides with the disappearance of connected pores, including primitive lines, as shown in Fig. 7. This results in more homogeneously distributed pores and significantly slower sintering.

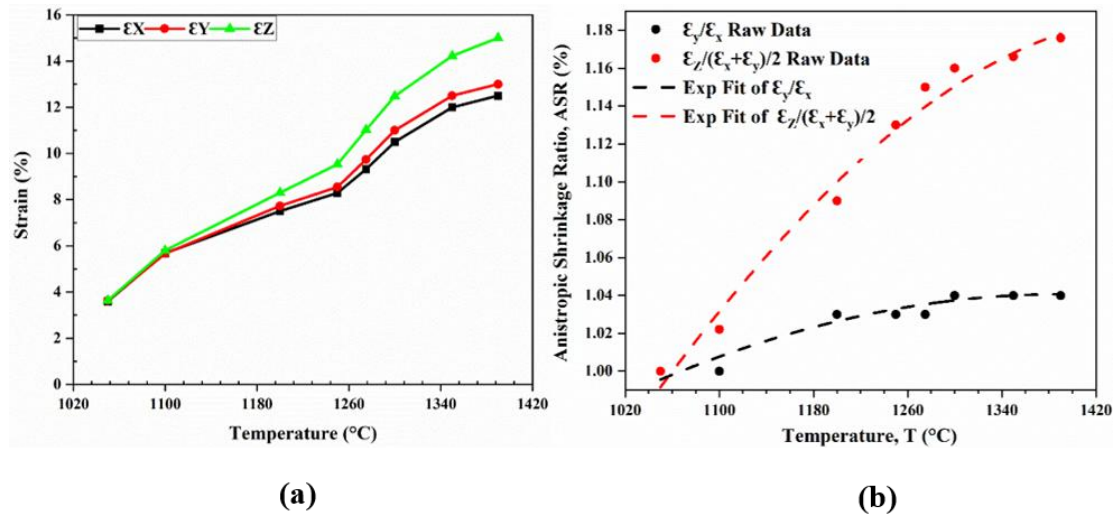


Figure 5-14 (a) Effect of sintering temperature and printing orientation on shrinkage strain along the ϵ_X – black, ϵ_Y – red, and ϵ_Z – green. (b) The influence of temperature on the in-plane (ϵ_Y/ϵ_X) and out-of-plane ($\epsilon_Z/((\epsilon_X + \epsilon_Y)/2)$) anisotropic shrinkage ratios.

Table 5.3 Fitted equations and R-squared values for the anisotropy data in (b).

	ϵ_Y/ϵ_X	$\epsilon_Z/(\epsilon_X + \epsilon_Y)/2$
Fitted Equation	$\text{ASR} = \exp(-0.689 + 1.06 \times 10^{-3} \times T - 3.8 \times 10^{-7} \times T^2)$	$\text{ASR} = \exp(-1.954 + 2.870 \times 10^{-3} \times T - 9.681 \times 10^{-7} \times T^2)$
R-Squared Value	0.943	0.986

Anisotropic sintering behavior is quantified within the powder bed plane (in-plane) and along the build direction (out-of-plane) in Fig. 14 (b). The in-plane anisotropy ratio (ASR_{IP}) is obtained with ϵ_Y/ϵ_X and the out-of-plane anisotropy ratio (ASR_{OP}) with $\epsilon_Z/((\epsilon_X + \epsilon_Y)/2)$, based on Fig. 14 (a). In both cases, the increase in anisotropic shrinkage follows the power law with equations provided in Table 3. The out-of-plane anisotropy is always more significant than in-plane anisotropy and increases sharply up to 1300 °C. For example, above 1250 °C, ASR_{OP} is 1.13 while ASR_{IP} is 1.03. This indicates a 13% higher anisotropic shrinkage out-of-plane along the build direction, attributed

to the presence of primitive lines in Fig. 7 (c). The reduced rate of change in ASR_{OP} observed above 1300 °C in Fig. 14 (b) coincides with the disappearance of primitive lines in Fig. 7 (c).

The negligible change in ASR_{IP} (1.00-1.04) suggests the shrinkage behavior on the powder bed plane is near quasi-isotropic. The slight difference in shrinkage ($\leq 4\%$) between the X- and Y-axes is likely due to the printhead. Fig. 15 demonstrates that sintered samples printed along the X-axis are slightly denser (3-4% more) than the sintered samples printed along the Y-axis when sintering between 1100 and 1200 °C. Lower porosity in the X-axis might be explained by greater interaction/compaction between the powder and binder particles along the printhead direction during the printing process and is largely dependent on parameters used during printing [138]. Nonetheless, the binder jetting GE series2 machine conditions are adequate to minimize anisotropy in the X-Y plane. This is further supported by similar microstructure evolution and pore alignment observed in the X-axis (Fig. 7a) and Y-axis (Fig. 7b) between 1100 °C and 1390 °C.

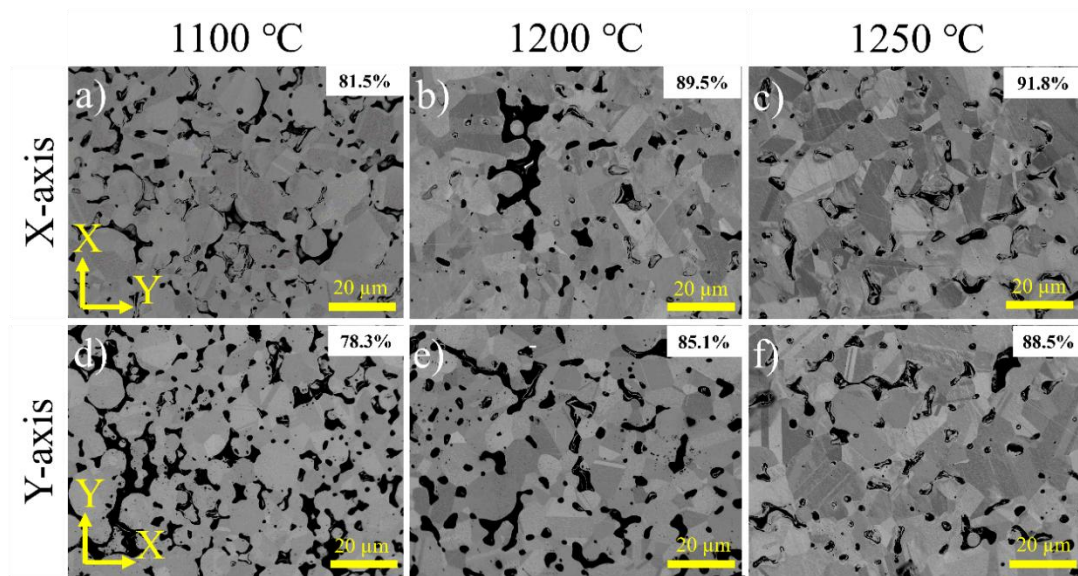


Figure 5-15 In-plane SEM images of BJ SS 316L sintered between 1100 °C and 1250 °C for the (a) X-axis and (b) Y-axis.

5.4.4 Effect of activation energy on anisotropic shrinkage

The anisotropic shrinkage behavior in Fig. 14b follows a continuous power law with no sharp change around 1250 °C observed in the maximum shrinkage in Fig. 14a. This means that the increase in shrinkage above 1250 °C occurs at a similar rate in all three printing orientations. Therefore, the increase in anisotropic shrinkage is not affected by the change in activation energy.

This is shown in Fig. 16, where the activation energies remain the same with varying printing orientations for all sintering temperatures. This results in a similar evolution of grain sizes (Fig. 13a), ferrite fractions (Fig. 10c), and bulk densities (Fig. 13b) for the X-, Y-, and Z-axes upon sintering.

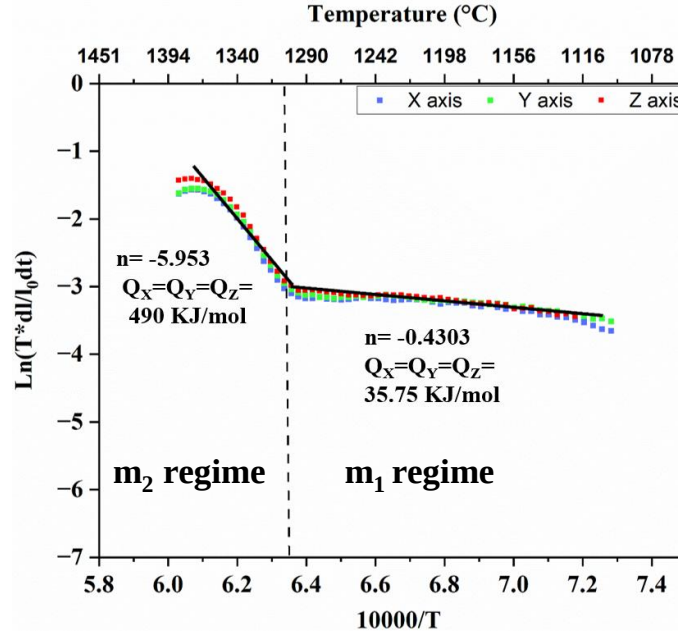


Figure 5-16 The effect of temperature on activation energy (Q) in different sintering shrinkage regimes, m_1 and m_2 , for the sample printed in the X-axis, Y-axis, and Z-axis sintered up to 1390 °C.

5.4.5 Effect of anisotropic pore distribution on sintering rate and viscous moduli

The quasi-isotropy sintering behavior observed in binder jetting coincides with the preferential alignment of pores perpendicular to the build direction, as shown in Fig. 7. This means that the atomic flux is expected to vary along different directions of the material during sintering. Since implementing finite element analysis is simpler than diffusion solutions, viscosity is more commonly used to predict the sintering behavior [164,165]. The viscosity function follows an Arrhenius law as follows:

$$\eta = \eta_0 \exp\left(-\frac{Q}{RT}\right), \quad (3)$$

where η_0 represents the pre-exponential factor for viscous flow, Q denotes the activation energy, T is the final sintering temperature, and R is the universal gas constant. In three dimensions, the viscosity pre-exponential factor (η_0) can be decomposed into three terms [164]:

$$\eta_x = \frac{-3 \gamma_s (1 - \theta)^2}{D \left[(1 - \theta)^2 \dot{\epsilon}_x + \left[\frac{2}{3} \frac{(1 - \theta)^3}{\theta} - \frac{1}{3} (1 - \theta)^2 \right] (\dot{\epsilon}_x + \dot{\epsilon}_y + \dot{\epsilon}_z) \right]}, \quad (4)$$

$$\eta_y = \frac{-3 \gamma_s (1 - \theta)^2}{D \left[(1 - \theta)^2 \dot{\epsilon}_y + \left[\frac{2}{3} \frac{(1 - \theta)^3}{\theta} - \frac{1}{3} (1 - \theta)^2 \right] (\dot{\epsilon}_x + \dot{\epsilon}_y + \dot{\epsilon}_z) \right]}, \quad (5)$$

$$\eta_z = \frac{-3 \gamma_s (1 - \theta)^2}{D \left[(1 - \theta)^2 \dot{\epsilon}_z + \left[\frac{2}{3} \frac{(1 - \theta)^3}{\theta} - \frac{1}{3} (1 - \theta)^2 \right] (\dot{\epsilon}_x + \dot{\epsilon}_y + \dot{\epsilon}_z) \right]}, \quad (6)$$

where γ_s is the surface energy, θ indicates the porosity, $\dot{\epsilon}$ stands for the strain rate, and D signifies diffusion. The strain-rates ($\dot{\epsilon}_x$, $\dot{\epsilon}_y$, $\dot{\epsilon}_z$) are proportional to the sintering rates shown in Fig. 11. Since the sintering rates vary along each major direction, the viscosity of the material is also expected to be anisotropic during the sintering of binder jetting green parts.

The sintering rates in Fig. 11 appear to be proportional to the pore density. Since there are more pores aligned perpendicular to the build direction, the sintering rates m_1 , m_2 , and m_3 are higher on the Z-axis compared to the X- and Y-axes in Fig. 11. This means that the diffusion rate is higher. The viscosity is lower along the Z direction compared to the two other directions (equation (4-6)). The relatively small difference between sintering rates along the X- and Y-axes confirms the quasi-isotropic behavior of the binder jet material discussed in section 4.2. The difference between the m_1 and m_2 sintering rates along the Z and X, Y directions increases with temperature. The m_1 rates reach a constant value at 1300 °C, above which the sintering rate m_2 in Fig. 11 (b) dominates the sintering. The gap between the Z-axis and X-, Y-axes continues to increase with temperature. During the isothermal section of the sintering cycle, the anisotropic shrinkage rate decreases with temperature, as shown in Fig. 11 (c). Below 1250 °C, the shrinkage rate is constantly higher along the Z-axis than the X- and Y-axes. This difference in shrinkage rate disappears above 1250 °C as there is negligible shrinkage in the isothermal section, as discussed in section 4.1.

5.5 Conclusion

The anisotropic sintering behavior in BJ 316L was studied. Six different sintering temperatures and three different printing orientations were considered, and the following key conclusions were drawn:

1. Two main temperature-dependent shrinkage regimes were observed during the sintering of BJ 316L parts. At lower temperatures (1100-1300 °C), low-intermediate shrinkage (6-13%) and relative density (76-93%) were governed by low activation energies (25-162 kJ/mol), promoting grain boundary diffusion. At higher temperatures (1300-1390 °C), high shrinkage (up to 15%) and density (up to 96%) were dictated by high activation energies (490-623 kJ/mol), promoting volume diffusion.
2. Significant shrinkage at elevated temperatures coincided with increased grain size and δ -ferrite formation observed in the sintered microstructure. The γ - δ interface accelerates the diffusion rate for pore coalescence, resulting in increased material densification.
3. The activation energies are not affected by the printing orientation. As a result, the microstructure of BJ 316L evolves similarly for all three printing orientations upon sintering. The grain size remains similar (23-32 μm) for low sintering temperatures and increases sharply (37-56 μm) above 1300 °C. Similarly, the ferrite fraction increases considerably (3-8%) at high temperatures. This results in similar sintered densities for all three printing orientations.
4. The shrinkage behavior on the powder bed is near quasi-isotropic, with very little anisotropy observed in the powder bed plane. The maximum difference in shrinkage between the X-axis and Y-axis is only 4%. This is attributed to greater interaction between the powder and binder particles along the printhead direction.
5. The out-of-plane anisotropic shrinkage is attributed to an anisotropic viscous sintering behavior. The primitive lines formed perpendicular to the build direction results in a higher diffusion rate and lower viscosity along the Z- axis than the X and Y- axes.
6. The out-of-plane anisotropic shrinkage follows the power law, and the increase in anisotropy is unaffected by the change in activation energy. The anisotropic shrinkage increases sharply (up to 16%) with sintering temperatures up to 1300 °C. Upon further

increase in sintering temperature, the decreased rate of anisotropic shrinkage coincides with the disappearance of primitive lines.

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CHAPTER 6 ARTICLE 2: Temperature and printing axis effects on anisotropic sintering in binder jetting additive-manufactured 17-4 PH stainless steel: Experimental and numerical study

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Abstract

The effects of sintering temperature and printing orientation on anisotropic shrinkage in binder-jetted 17-4 PH stainless steel are investigated using a combined experimental and finite-element modeling approach. Quasi-isotropic shrinkage is observed in the X–Y plane, while the Z-direction shows an anisotropic shrinkage ratio (ASR) up to 1.4, mainly due to primitive line collapse above 1250 °C when Grain boundary diffusion ($Q = 100\text{--}125$ kJ/mol) shifted to volume diffusion ($Q = 250\text{--}320$ kJ/mol). A similar activation energy was observed along the X, Y, and Z axes. However, the transition temperature (1250 °C) is slightly higher than what is normally reported for conventional powder metallurgy (1000–1150 °C) due to carbon residue from the binder. Finite element simulations show higher shear ($G = 12000$ MPa · s) and bulk ($K = 26400$ MPa · s) modulus along the Z axis compared to the powder bed plane due to the directional pore distribution. As the temperature increases, pores become more homogenously distributed, leading to more isotropic shrinkage mechanisms and similar K and G values across the powder bed.

Keywords: Binder jetting, Anisotropy sintering, SS 17-4 PH, Viscoplastic sintering model

6.1 Introduction

Binder jetting (BJ) additive manufacturing (AM) is a rapidly advancing technology that enables the production of complex and near-net-shape metal parts through a layer-by-layer approach at room temperature [1,2]. A broad range of metals, such as stainless steel [130,166–168], nickel [118–122], aluminum [123,169], Cobalt-chrome [170], and magnesium [126,127] can be processed

using BJAM. This method offers several advantages, including high productivity, the capability to process a wide range of materials, and reduced material waste compared to traditional manufacturing techniques [55]. Among the materials commonly used in BJ, stainless steel 17-4 PH is particularly valuable due to its high strength, excellent corrosion resistance, and precipitation-hardening characteristics, making it suitable for applications in aerospace, automotive, and medical industries [3,4,25,34,92,171–174].

Printed (green) parts require sintering, a heat treatment process essential for achieving fully dense, high-strength components with precise dimensional accuracy and optimized mechanical-structural properties [3,4,137]. However, sintering introduces significant challenges, including material deformation and anisotropic shrinkage, which can severely limit the dimensional accuracy and geometric precision of the final part [138]. Anisotropic sintering shrinkage can lead to a reduction in the final part dimensions by as much as 20 %, primarily due to non-uniform dimensional changes [139,167,175]. This dimensional reduction is mainly driven by inherent irregularities in green parts, such as the presence of primitive lines, variations in porosity, and inconsistencies in binder distribution, all of which contribute to anisotropic shrinkage behavior [140]. The irregularities are predominantly caused by the operational mechanics of the printing process [141] such as powder's particle size distribution (PSD), morphology, spreading dynamics, printhead movement, jetting behavior, and binder saturation levels [143]. These irregularities influence the density and binding strength, modifying green part properties in different orientations [139,142,143]. Mostafaei et al. [176] showed a strong variation in the viscous shear modulus (G) and bulk modulus (K) of the green part with temperature and green part orientation, demonstrating their critical role in predicting the dimensional change and densification kinetics during sintering.

Phase transformation is another important factor that contributes significantly to the anisotropic sintering behavior in the BJ process. Esmati et al. [175] highlighted the impact of δ -ferrite precipitation on the sintering kinetics and densification behavior of BJ-316L. At higher temperatures (>1300 °C), δ -ferrite enhances material diffusivity, thereby accelerating densification and reducing porosity. Austenite-ferrite phase transformation shifts the dominant sintering mechanism from grain boundary diffusion at lower temperatures (1100–1300 °C) to volume diffusion at higher temperatures, significantly increasing the anisotropic shrinkage up to 18 %. Elemental composition, with its influence on microstructure evolution and diffusion mechanism, is important in controlling sintering behavior and dimensional accuracy. Rios et al. also [87]

concluded that an increase in sintering temperature accelerates δ -ferrite precipitation, coupled with elemental composition, enhances sintering-induced anisotropy. Wu et al. [177] comprehensively investigated and brought out the influential role of residual carbon level on phase formation and shrinkage of BJ-17-4 PH during sintering.

Anisotropic sintering in binder jet stainless steel has been taken up well by previous researchers; however, most of the research has been focused on 316L grade for a single sintering temperature [13,87,142,144], thereby leaving a significant gap in establishing a temperature-dependent mechanism. Moreover, anisotropic sintering has been limitedly explored for 17-4 PH stainless steel, and no comprehensive study about it is available in the literature. There appears to be a pressing need to expand the current understanding of BJ-17-4 PH's anisotropic shrinkage, taking into consideration the phase transformation and densification kinetics phenomenon across a wider range of sintering temperatures.

This study aims to fill the existing gaps in the literature, making a novel attempt to establish the effect of multiple sintering temperatures, printing orientation, and microstructure evolution on anisotropic sintering shrinkage behavior in 17-4 PH binder jet fabricated parts. A comprehensive experimental-numerical framework has been proposed to investigate anisotropic sintering shrinkage behavior for refining shrinkage models and ensuring dimensional accuracy. The experimental analysis is complemented by finite element simulations to explain the underlying mechanisms of anisotropic sintering shrinkage and densification, including interlayer bonding, pore morphology, and phase evolution. This integrated approach provides a comprehensive understanding of how process parameters influence shrinkage behavior in binder jetting. Microstructural characterization, dilatometry, and finite element modeling are used to evaluate the effects of sintering temperature and printing axis on dimensional shrinkage. Samples printed in X, Y, and Z directions are analyzed for in-situ shrinkage, grain size, porosity, and phase transformations. An analytical model for anisotropic sintering is also implemented to investigate the role of pore distribution and direction-dependent viscosity in driving anisotropic deformation behavior in binder-jetted 17-4 PH stainless steel.

6.2 Experimental procedures

6.2.1 Materials

In this study, gas-atomized 17-4 PH stainless steel powder was used. Fig. 1(a) displays the powder morphology characterized mainly by spherical particles with some smaller satellite particles adhered to their surfaces. The particle size distribution (PSD), measured by the CAMSIZER X2 system (RETSCH, Wuppertal, Germany), includes D10 at 5.1 μm , D50 at 10.2 μm , and D90 at 20 μm , as shown in Fig. 1(b). The elemental composition of the 17-4 PH powder is detailed in Table 1.

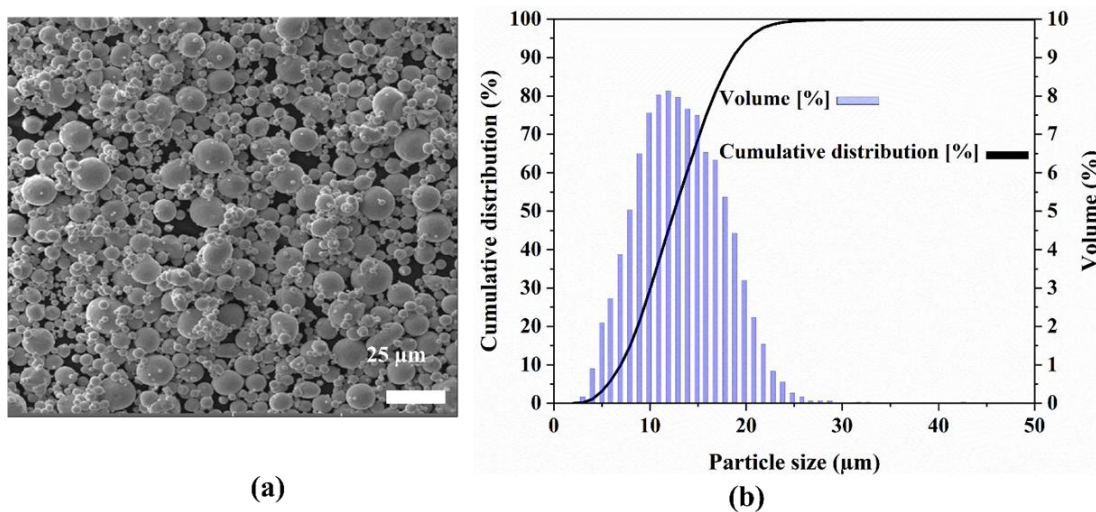


Figure 6-1 (a) SEM micrograph and (b) PSD of gas-atomized SS 17-4 PH powder.

Table 6.1. Elemental compositions of 17-4 PH powder in wt.%.

Fe	Cr	Ni	Cu	Mn	Si	Nb	N	O	C	P	S
Bal.	16.3	4.4	4.2	0.4	0.4	0.19	0.07	0.05	0.026	0.034	0.03

6.2.2 Binder jetting

Cubical samples ($10 \times 10 \times 20 \text{ mm}^3$) of 17-4 PH powder were printed in X, Y, and Z axes using an Exone series Innovent+ binder jet system (Fig. 2). The X, Y, and Z axes represent the recoater, printhead movement, and build directions. Table 2 presents the binder jetting parameters used in the study. A commercial water-based binder system (ExOne AquaFuse) was used for printing. This binder consists primarily of a glycol-based carrier with proprietary additives to promote adhesion

and flowability [178]. Following printing, the green parts were cured in a convection oven at 180 °C for 6 hours to allow the binder to fully cure and provide sufficient mechanical integrity [25]. Post-curing, the printed specimens were identified as green parts. A geometrical approach was used to measure the volume and weight of the green part, thereby obtaining green density.

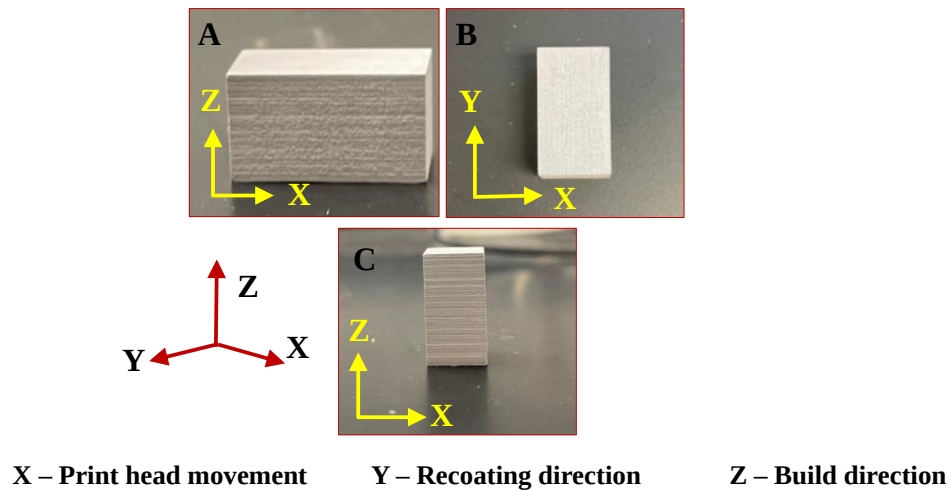


Figure 6-2 Images of binder-jetted 17-4 PH stainless steel samples printed along three orthogonal directions: (A) X-axis (recoater direction), (B) Y-axis (printhead direction), and (C) Z-axis (build direction).

Table 6.2 Binder jet printing parameters

Printing parameters	Value
Binder set time (s)	20
Binder saturation (%)	80
Recoating speed (mm/s)	80
Bed temperature (°C)	55
Layer thickness (μm)	50

6.2.3 Dilatometry sintering test

The dilatometry sintering process used in this study was designed based on thermogravimetric analysis (TGA) of the green part, supplemented by dilatometry experiments and prior studies [179–181] on binder jetting of 17-4 PH stainless steel.

To determine the appropriate debinding conditions for binder removal, thermogravimetric analysis (TGA) was conducted on the green parts as shown in Fig. 3(a). The curve reveals a negligible weight loss up to ~ 300 °C, followed by a gradual decline and a sharp drop in weight between 400 °C and 500 °C, which corresponds to the binder's thermal degradation and volatilization. Based on the TGA curve and prior studies [175,179,182], the debinding cycle was designed in three stages, as illustrated in Fig. 3(b): (1) ramp to 200 °C at 5 °C/min with a 90-minute hold, (2) slow heating between 400–600 °C for 7 hours, and (3) pre-sintering at 800 °C for 1.5 hours. This ensured complete binder burnout, providing initial structural strength before the sintering.

The sintering temperatures selected for this study ranged from 1100 to 1350 °C, which was determined based on differential scanning calorimetry (DSC) data, dilatometry results, and prior research on sintering of 17-4 PH stainless steel [91,110,179–181]. These temperatures were chosen to capture the grain boundary diffusion-volume diffusion transition and systematically investigate the effects of temperature on shrinkage, microstructure, and anisotropy shrinkage during sintering. Dilatometry test was performed in an Ar–5 % H₂ atmosphere, maintained at a constant gas flow rate of 100 mL/min. Fig. 3(c) illustrates the schematic of the heat treatment cycle used during dilatometry sintering. A three-step sintering process consists of heating to sintering temperature at a constant rate of 5 °C/min, followed by a dwell time of 4 hrs and then cooling down to room temperature at 10 °C/min.

Dilatometric sintering was conducted at six different temperatures, ranging from 1100 °C to 1350 °C, for all samples, as summarized in Table 3. To verify repeatability, each temperature and printing orientation was tested twice. The final density of the samples after dilatometric sintering was measured using the Archimedes method.

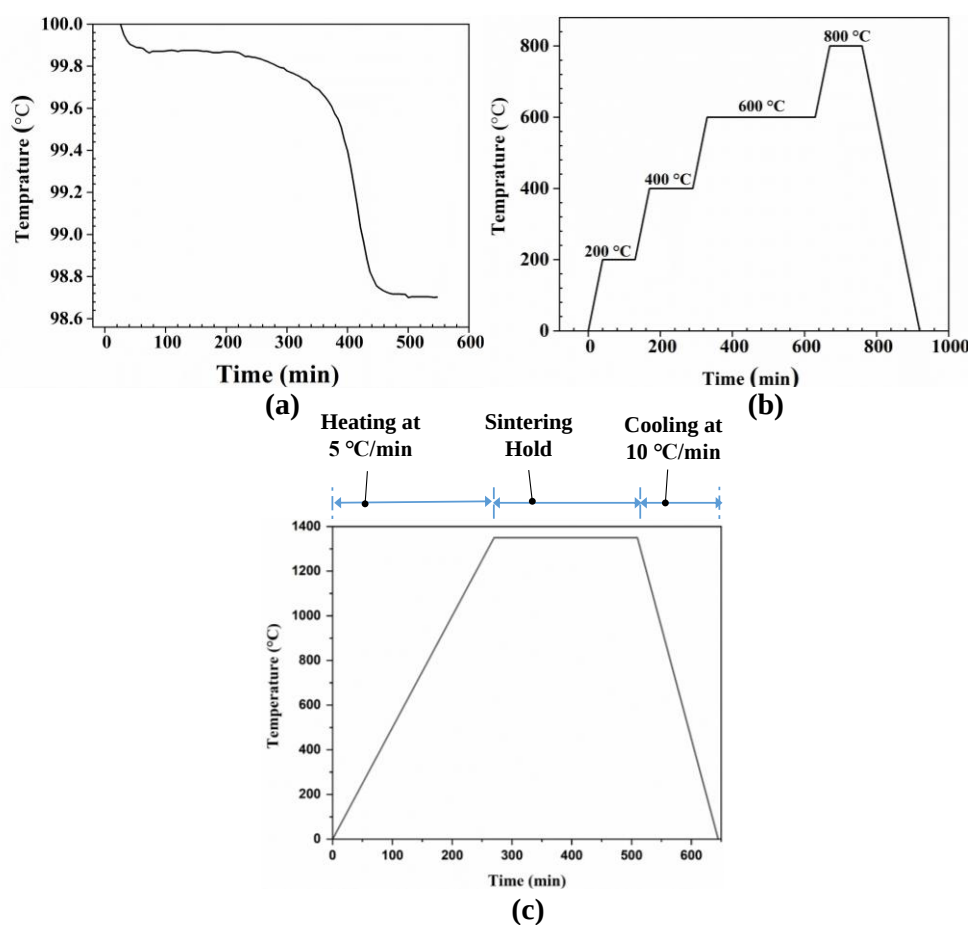


Figure 6-3 (a) TGA curve of the green part showing thermal decomposition of the binder. (b) Multistep debinding profile under Ar–5 % H₂ atmosphere (c) The heating cycle in dilatometry sintering of BJ SS17-4 PH.

Debonded samples were placed in a Netzsch DIL 402S dilatometer with the longitudinal axis aligned parallel to the pushrod with a contact force of 3 mN to perform sintering. In-situ shrinkage along the printing axes (X, Y, and Z) was measured during the sintering process.

Linear shrinkage was measured as the percentage reduction in sample length during the sintering process, calculated from the dilatometer displacement data relative to the initial green length. The shrinkage rate, on the other hand, refers to the rate of change in shrinkage with respect to time and was obtained as the first derivative of the shrinkage curve. These parameters are used to evaluate densification behavior and are discussed further in the results section.

Table 6.3 Dilatometry sintering parameters of BJ SS17-4 PH.

Dimensions (X,Y,Z)		Number of samples dilatometry sintered		
Final Temperature (°C)	Holding Time (Hour)	Orientation (X)	Orientation (Y)	Orientation (Z)
1100	4	2	2	2
1150	4	2	2	2
1200	4	2	2	2
1250	4	2	2	2
1300	4	2	2	2
1330	4	2	2	2
1350	4	2	2	2

6.2.4 Microstructure characterization

Microstructural analysis of the pre- and post-sintered specimens was performed using optical and scanning electron microscopy (OM/SEM). To investigate the influence of print direction and sintering temperature on microstructural evolution, the specimens were cut along the printing orientation. The test samples were initially ground using abrasive papers of varying grit sizes (320, 600, and 1200) to obtain a smooth surface suitable for analysis. They were subsequently polished with a diamond suspension followed by 0.04 μm colloidal silica. Polished samples were etched with Kalling's reagent (33 mL H_2O , 33 mL ethanol, 33 mL HCl , and 1.5 g CuCl_2) for 120 seconds.

To assess phase distribution (ferrite and martensite), porosity, and grain size, the microstructural analysis was performed using a Keyence VHX7000. The average grain size was measured from optical micrographs using the line intercept method, as specified in ASTM E12–13 [148]. Ferrite volume fraction was determined through image analysis using ImageJ software. Further microstructural and phase characterization was performed using a JEOL JSM-7600F field-emission scanning electron microscope (FEG-SEM) coupled with an Oxford Instruments X-Max 80 mm EDS detector. Phase identification and quantification in the microstructure were performed using X-ray diffraction (XRD) with a Bruker D8 Advance system equipped with Cu K α radiation. The differential scans were obtained using a 40 kV/40 mA setting in the 2θ range of 30° to 90° with a step size of 0.65° . The obtained scans were analyzed using DIFFRAC. EVA software, combined with the ICDD-PDF4+ database, for phase identification and quantification.

6.2.5 Theoretical modelling

This study utilized the fully implicit constitutive model for sintering developed by Olevsky et al. [183]. The model predicts sintering-induced shrinkage during binder jetting by integrating porosity and temperature-dependent properties, as well as anisotropic sintering stress expressions, which drive the anisotropic behavior. By validating the model-predicted shrinkage behavior against experimental data, the model is able to predict shear and bulk viscous moduli, thereby improving the understanding and control of sintering behavior in binder jetting. For calculating porosity and deformation, the constitutive law is presented in equation (1).

$$\dot{\epsilon}^{vc} = \frac{\sigma'}{2G_p} + \frac{\sigma_m - \sigma_s}{3K_p} \mathbf{I} \quad (1)$$

Where $\dot{\epsilon}^{vc}$ is the viscoplastic creep strain rate, σ' is the deviatoric stress tensor, σ_m is the hydrostatic stress, σ_s is the sintering stress, $\mathbf{I}$ is the identity tensor, G_p and K_p are the shear and bulk viscosities, respectively.

The overall strain rate ($\dot{\epsilon}$) is broken down into three components: the viscous creep strain rate ($\dot{\epsilon}^{vc}$), thermal strain rate ($\dot{\epsilon}^T$), and elastic strain rate ($\dot{\epsilon}^e$), as shown in equation (2):

$$\dot{\epsilon}_{(t+1)} = \dot{\epsilon}_{t+1}^{vc} + \dot{\epsilon}_{t+1}^T + \dot{\epsilon}_{t+1}^e \quad (2)$$

The thermal strain depends solely on temperature changes and is determined independently using the viscous creep and elastic strains. The viscous creep and elastic strain rate variables were considered zero at the first iteration and were calculated through iterations. At the start of each time step, the porosity ($\theta_{(t+1)}$) is the same as the previous iteration ($\theta_{(t)}$). The viscous creep strain rate is influenced by both the applied and internal sintering stresses, and it varies with grain size and the current porosity. For calculating the sintering stress ($\sigma_{s(t+1)}$) equation (3) was used:

$$\sigma_{s(t+1)} = \frac{6\omega}{r_{g(t+1)}} (1 - \theta_{t+1})^2 \quad (3)$$

Where ω is the surface tension, $r_{g(t+1)}$ is the current grain size, and θ_{t+1} is the current porosity.

A sigmoid was developed to modify the sintering stress relation given in equation (3). The modified sintering stress σ_s^{mod} takes the following form:

$$\sigma_s^{mod} = \sigma_s \left(\sigma_{sl} + \frac{(1 - \sigma_{sl})}{1 + e^{\frac{TEMP - T_{st}}{D_{ss}}}} \right) \quad (4)$$

Equation (4) was used to control the sintering stress to have a better fit with the experimental results. σ_{sl} is the lower limit of the sigmoid, T_{st} is the transition temperature for the sintering stress, D_{ss} is the value used to spread out the sigmoid. For this study, sintering stress was considered to be only due to the elastic deformation. The sintering stress rate ($\dot{\sigma}_{t+1}$) was formulated as (equation (5)):

$$\dot{\sigma}_{t+1} = L_{t+1}^e \dot{\epsilon}_{t+1}^e \quad (5)$$

$L_{(t+1)}^e$ is the isotropic elasticity tensor which depends on temperature and porosity. For the calculation of $\dot{\epsilon}_{t+1}^{vc}$ equation (6) was used:

$$\dot{\epsilon}_{t+1}^{vc} = \nu_{t+1} [\sigma_{t+1} + \sigma_{s(t+1)} \delta_{ij}] \quad (6)$$

In equation (6), the viscosity tensor $v_{(t+1)}$ varies with the bulk viscosity ($K_{(t+1)}^{vc}$) and shear viscosity ($G_{(t+1)}^{vc}$) and is given as

$$V = \begin{bmatrix} \left(\frac{1}{9K_{(t+1)}^{vc}} + \frac{1}{3G_{(t+1)}^{vc}}\right) & \left(\frac{1}{9K_{(t+1)}^{vc}} - \frac{1}{6G_{(t+1)}^{vc}}\right) & \left(\frac{1}{9K_{(t+1)}^{vc}} - \frac{1}{6G_{(t+1)}^{vc}}\right) & 0 & 0 & 0 \\ \left(\frac{1}{9K_{(t+1)}^{vc}} - \frac{1}{6G_{(t+1)}^{vc}}\right) & \left(\frac{1}{9K_{(t+1)}^{vc}} + \frac{1}{3G_{(t+1)}^{vc}}\right) & \left(\frac{1}{9K_{(t+1)}^{vc}} - \frac{1}{6G_{(t+1)}^{vc}}\right) & 0 & 0 & 0 \\ \left(\frac{1}{9K_{(t+1)}^{vc}} - \frac{1}{6G_{(t+1)}^{vc}}\right) & \left(\frac{1}{9K_{(t+1)}^{vc}} - \frac{1}{6G_{(t+1)}^{vc}}\right) & \left(\frac{1}{9K_{(t+1)}^{vc}} + \frac{1}{3G_{(t+1)}^{vc}}\right) & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1}{G_{(t+1)}^{vc}} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1}{G_{(t+1)}^{vc}} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{G_{(t+1)}^{vc}} \end{bmatrix} \quad (7)$$

The strain rate partitioning was repeated until the viscous creep strain rate converged. After each iteration, the deformation-induced porosity was updated using a secondary loop. The reduction in porosity was considered the primary mechanism for accommodating viscous creep strain. When the creep strain rate was calculated, the porosity rate ($\dot{\theta}_{t+1}$) was determined using the following equation:

$$\dot{\theta}_{t+1} = (1 - \theta_{t+1})(\dot{\epsilon}_{(t+1)11}^{vc} + \dot{\epsilon}_{(t+1)22}^{vc} + \dot{\epsilon}_{(t+1)33}^{vc}) \quad (8)$$

At the end of the current time step, the porosity was calculated using the data from the previous time step as follows:

$$\theta_{t+1} = \theta_t + \Delta t \dot{\theta}_{t+1} \quad (9)$$

After that, the material properties that depend on porosity were recalculated accordingly. The viscosity of the SS 17-4 PH was calculated from equation (10) [184]

$$\eta_{t+1} = \eta_{0(t+1)} e^{\frac{Q_v(t+1)}{RT_{t+1}}} \quad (10)$$

Where $\eta_{0(t+1)}$ is the viscosity coefficient, Q_v is the viscosity activation energy, R is the gas constant, and T is the absolute temperature.

The equation for sigmoidal high and low-temperature activation energy is used to compare numerical and experimental data, which is defined as:

$$Q_{v(t+1)} = Q_{vh} + \frac{Q_{vl} - Q_{vh(t+1)}}{1 + \frac{e^{T_{t+1} - T_{critiv1}}}{20}} \quad (11)$$

In this expression, Q_{vh} and Q_{vl} denote the high- and low-temperature activation energies for viscosity, respectively. The transition between these two regimes occurs at the critical temperature ($T_{critiv1} = 970$ °C), which was determined from experimental data illustrated in Fig. 5(a). This temperature corresponds to the onset sintering temperature, and equation 8 governs the shift from no sintering to the onset of grain boundary diffusion.

The factor of 20 in equation (11) controls the range of temperature over which the transition occurs between low and high activation energies. This range is essential for capturing the results observed during experiments. The high-temperature activation energy is also related to the temperature and causes a transition between grain boundary diffusion (Q_{vgb}) and volume diffusion (Q_{vv}).

$$Q_{vh(t+1)} = Q_{vv} + \frac{Q_{vgb} - Q_{vv}}{1 + \frac{e^{T_{t+1} - T_{critiv2}}}{20}} \quad (12)$$

Where $T_{critiv2} = 1250$ °C is the grain boundary to volume diffusion transition temperature derived from experimental results presented in Fig. 13.

The viscosity coefficient depends on the active diffusion mechanism and is defined as:

$$\eta_{0(t+1)} = \eta_v + \frac{\eta_v - \eta_{gb}}{1 + \frac{e^{T_{t+1} - T_{critiv2}}}{20}} \quad (13)$$

The viscosity can be calculated in all Cartesian coordinates of X, Y, and Z as :

$$\eta_{(t+1)x} = \eta_{0x(t+1)} e^{\frac{Q_{v(t+1)}}{RT_{t+1}}} \quad (14)$$

$$\eta_{(t+1)y} = \eta_{0y(t+1)} e^{\frac{Q_{v(t+1)}}{RT_{t+1}}} \quad (15)$$

$$\eta_{(t+1)z} = \eta_{0z(t+1)} e^{\frac{Q_{v(t+1)}}{RT_{t+1}}} \quad (16)$$

Where η_v and η_{gb} represent the viscosity coefficients for volume diffusion and grain boundary diffusion, respectively.

The bulk and shear viscosities moduli, which depend on porosity, were used from the model proposed by Olevsky [185] and were computed accordingly:

$$K_{t+1}^{vc} = \frac{4(1 - \theta_{t+1})^3}{3\theta_{t+1}} \eta_{t+1} \quad (17)$$

$$G_{t+1}^{vc} = (1 - \theta_{t+1})^2 \eta_{t+1} \quad (18)$$

It was assumed that the variation of viscosity is linear. The viscous creep strain rate was calculated using the moduli and the viscosity tensor.

Since grain size significantly influences sintering stress, modeling grain growth is crucial. Experimental studies [186,187] have demonstrated that grain growth in stainless steels exhibits both high- and low-temperature activation energies. To continuously capture this temperature-dependent behavior, the activation energy is expressed through a sigmoidal function:

$$Q_{g(t+1)} = Q_{gh} + \frac{Q_{gl} - Q_{gh}}{1 + \frac{e^{T_{t+1} - T_{critig}}}{20}} \quad (19)$$

Here, $Q_{gh} = 320$ kJ/mol represents the activation energy at elevated temperatures, while $Q_{gl} = 125$ kJ/mol corresponds to the value at lower temperatures. The critical transition temperature (T_{critig}) is 1250 °C. All these values were obtained from the experimental results shown in Fig. 13 and align with the observations reported by Kashyap and Tangri [188].

The following equation is used to calculate grain growth.

$$\dot{r}_{g(t+1)} = \frac{Ae^{-\frac{Q_{g(t+1)}}{RT_{t+1}}}}{r_{g(t)}} \quad (20)$$

In the equation (20), A denotes the pre-exponential factor associated with grain growth, $r_{g(t)}$ represents the grain size at the previous time step, and R is the universal gas constant. As the green density of BJAM is relatively lower than that of other processes, it is assumed that grain growth is primarily due to the diffusion mechanism [189].

As a result, the coefficient 'A' is a function of T_{critv2} . Using an explicit integration, for temperatures below T_{critv2} , the coefficient 'A' is for grain boundary diffusion, while for those above T_{critv2} , it is for volume diffusion.

The elasticity (E) and Poisson's ratio (ν) of SS 17-4 PH, which vary with both porosity and temperature, are determined using an empirical model developed by [188] given:

$$E = 199510 - 65.63T_{t+1} - 0.00267T_{t+1}^2 - 1.75 \times 10^{-6}T_{t+1}^3(1 - \theta_{t+1}^{9.95}) \quad (21)$$

$$\nu = 0.238 - 3.95 \times 10^{-5}T_{t+1} + 9.17 \times 10^{-8}T_{t+1}^2(1 - \theta_{t+1}^{1.51}) \quad (22)$$

In every iteration, a separate subroutine was used to calculate the porosity. After that, all material properties dependent on porosity were recalculated to verify the convergency. The formulation for the tangent modulus used in LS-DYNA is from the work done by Al-Qudsi et al. [190]. They validated the variations in tangent modulus for different sintering models. For the viscosity formulation, a model derived from Olevsky's [185] was used.

A three-dimensional finite element simulation of the sintering process was done using eight-node hexahedral elements. The displacements in the Z direction for all nine nodes on the bottom face were constrained to zero. Movement in the X and Y directions is restricted for one node in the corner of the bottom face. Additionally, the corner node aligned in the Y-direction from the fully constrained node was restricted from moving in the X-direction. This setup prevents rotations in 3-D without constraints on the geometry's free sintering.

A uniform temperature distribution was applied to the nodes on the bottom surface that were restricted from displacement in the Z-direction. Values were taken from Table 3. Given the sample sizes and prolonged hold times, the effects of heat transfer during the sintering process are negligible. The simulations utilized a customized material model defined in LS-DYNA. An automatic adaptive time-stepping method was employed. It begins with an initial time step of 100 seconds and can be increased to a maximum of 1000 seconds.

6.2.6 Model calibration procedure

The model described above was individually calibrated for the X and Z directions to evaluate the effect of pore directionality inside the green specimens on material behavior during sintering.

Esmati et al. [130,175] demonstrated that the dilatometer shrinkage exhibits a transversely isotropic behavior, characterized by near-uniform shrinkage on the X-Y plane of the material; therefore, only one of these directions is sufficient to approximate the shrinkage behavior in that plane. The viscosity coefficients for volume (η_v) and grain boundary (η_{gb}) diffusion for each of the two directions considered (X and Z) was calibrated using the dilatometer strains for the respective directions. The viscosity constants for volume (η_v) and grain boundary (η_{gb}) diffusion from equation (13) was calibrated using dilatometer strain results on a temperature-by-temperature basis to determine the evolution of the bulk and shear viscous moduli.

6.3 Results

6.3.1 Green density

The printed specimens in different orientations exhibited consistent green density (58–60 %) and green strength (6–8 MPa). However, pore distribution was influenced by the direction of printing. As shown in Fig. 4, the surface morphology of 17-4 PH stainless steel samples fabricated along the X, Y, and Z directions reveals pores typical of green parts. For samples printed in the X and Y directions, pores appeared to be randomly distributed. In contrast, those printed along the Z-axis displayed a periodic pore pattern aligned with primitive lines oriented normal to the build direction (BD). This regularity in the Z-direction is a characteristic feature of the binder jetting (BJ) process, which is in agreement with similar observations made by previous researchers [149,150,175].

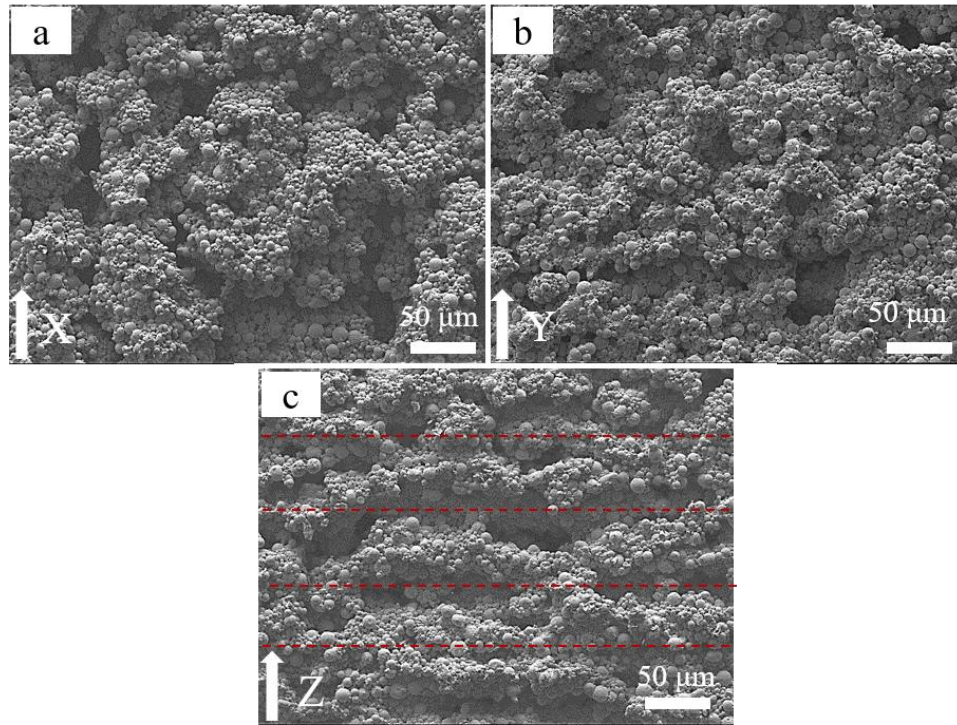


Figure 6-4 SEM micrographs of green specimens printed in the (a) X-, (b) Y-, and (c) Z-directions. In (c), the primitive lines are normal to the Z-axis.

6.3.2 Dilatometry sintering

Sintering stages

Fig. 5 illustrates the thermal profile, shrinkage behavior, and shrinkage rate of binder-jetted 17-4 PH stainless steel samples sintered along the Z-axis at a peak sintering temperature of 1330 °C. Fig. 5(a) presents the heating profile (blue curve) and corresponding sintering shrinkage curve (black curve) as a function of time. The heating profile includes a gradual rise in temperature up to the sintering temperature of 1330 °C, followed by an isothermal hold between t_s and t_f , which define the beginning and end of the dwell time, respectively. The dilatometry curve shows the shrinkage over time. The shrinkage curve indicates that from $t=0$ to $t=t_0$ (around 200 minutes), the sample undergoes thermal expansion, reaching a maximum of approximately 1 %. This expansion occurs as the temperature rises to about 970 °C, corresponding to the sintering onset temperature. After t_0 (onset sintering time), shrinkage begins, and the curve shows a continuous decrease in length, reflecting the material's densification during the sintering process.

Fig. 5(b) shows the shrinkage rate (black curve), defined as the first derivative of the shrinkage data from Fig. 5(a), alongside the same heating profile. This figure shows three different m_1 , m_2 , and m_3 slopes marked in pink, cyan, and orange, respectively. These slopes show changes in the sintering shrinkage rate. A small peak (red circle) in the shrinkage rate curve (Fig. 5(b)) represents the volume change associated with the martensite to austenite transformation [92,179]. In a short period from t_0 (200 min) to t_s (270 min), the sintering rate changes rapidly and is specified by m_1 and m_2 . After this, in an isothermal stage from the t_s to t_f , the shrinkage rate is constant and represented by m_3 .

The effect of sintering temperature on the %shrinkage shows an interesting trend in the X, Y, and Z axes (Fig. 6). The shrinkage observed in the X (Fig. 6a) and Y (Fig. 6b) directions is relatively similar, whereas the Z-direction (Fig. 6c) displays significantly greater shrinkage. For example, at a sintering temperature of 1330 °C, the shrinkage along the X and Y axes reaches 12.30 % and 12.73 %, respectively, while the Z-axis shows a notably higher value of 16.68 %.

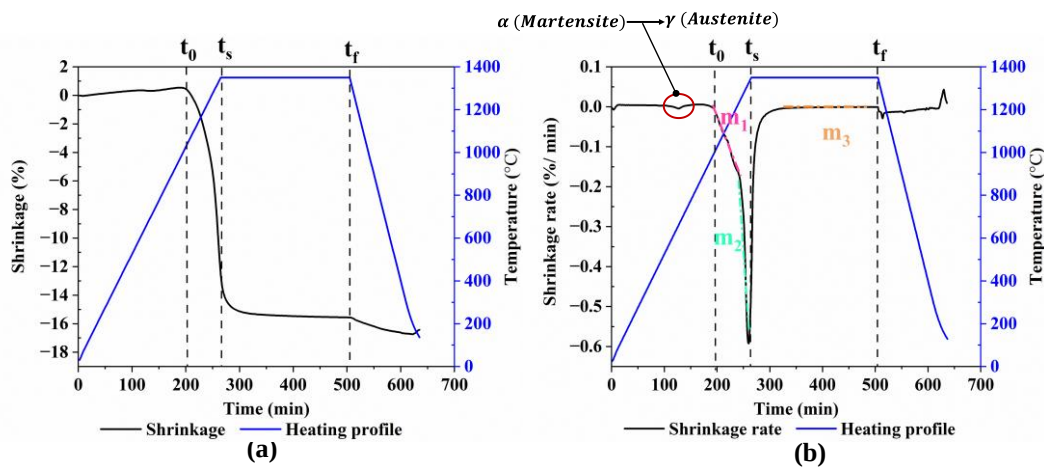


Figure 6-5 Heating profile and dilatometry curves: (a) Shrinkage percentage vs. time, (b) Shrinkage rate vs. time for different sintering regimes.

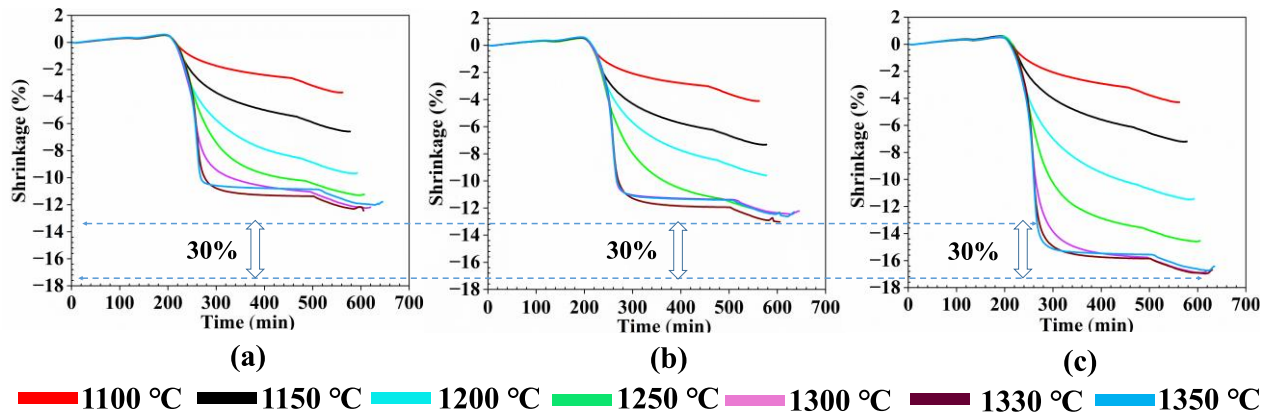


Figure 6-6 Linear shrinkage curves at different sintering temperatures for samples printed along (a) X-, (b) Y-, and (c) Z-axis.

6.3.3 Effect of sintering temperature on microstructure

SEM images of the binder-jetted samples provide insights into changes in solid content and pore structure under various sintering temperatures (Fig. 7). Across all printing directions, the solid volume fraction increases progressively with temperature, reaching a maximum sintered density of around 97 % at 1330 °C. As the temperature rises, pores transition from irregular and interconnected to more spherical and isolated forms. Between 1100 °C and 1250 °C, pore connectivity and alignment are still evident in all orientations. In samples printed along the X and Y directions, pores tend to align vertically, following the printing axis (see red dashed lines in Fig. 7(a) and 7(b)), while in the Z-direction, pores appear horizontally aligned, normal to the build direction (Fig. 7(c)). In the BJ process, the impact of high-velocity binder droplets on the powder bed transfers momentum to the powder particles, causing their ejection and forming primitive lines [24,149]. In the present work, the Z-axis printed samples exhibited the highest shrinkage, up to 17 % (Fig. 6c), which is attributed to the collapse of primitive lines aligned perpendicular to the build direction.

To gain deeper insight into the metallurgical factors influencing sintering shrinkage, high-magnification SEM images were analyzed for samples printed along the X, Y, and Z orientations (Fig. 8). Additionally, the phase and compositional analysis of the sintered sample's microstructure was done using XRD (Fig. 9) and EDS (Fig. 10) techniques. The microstructure is sensitive to sintering temperature for all printing orientations. The porosity level in Fig. 8 is comparatively higher at a lower sintering temperature of 1100 °C, with open pores appearing in irregularly shaped

morphologies. This irregularity is mainly due to incomplete consolidation of powder particles and limited particle bonding [151]. XRD analysis (Fig. 9) at this temperature shows a microstructure primarily composed of martensitic laths, retained austenite, and uniformly distributed porosity. At an elevated sintering temperature of 1200 °C, pores are predominantly located at triple grain boundary junctions, and the presence of a secondary phase (δ -ferrite, indicated by red arrows) becomes noticeable. By 1250 °C, most pores are reduced to small, isolated features within the grains, the retained austenite phase is no longer observed (Fig. 9), and the amount of secondary phase increases. This increase corresponds to the formation of chromium-rich and nickel (Ni) depleted regions approximately 30 μm in size, characteristic of delta ferrite (δ -ferrite) in 17-4 PH stainless steel. δ - ferrite tends to precipitate around isolated pores, preferring to nucleate near porosity because most porosity is located along the original interparticle boundaries. These heterogeneities provide optimal sites for ferrite nucleation from austenite. The BJ 17-4 PH microstructure in Fig. 8 shows that the δ -ferrite phase is predominantly present in thick lath or skeletal morphology. During sintering at 1300 °C, the microstructure shows a noticeable presence of δ - ferrite, which begins to form a network surrounding the martensite structure. Porosity is reduced compared to lower temperatures, with isolated pores resulting in a densification of approximately 96 %. At the higher temperature of 1330 °C (Fig. 8), the delta ferrite network becomes more pronounced and fully developed, enclosing the martensite structure more clearly. The ferrite regions increase in size due to enhanced atomic mobility, while porosity is nearly eliminated, leading to densification exceeding 97 %.

The EDS elemental maps (Fig. 10) further reveal the elemental distribution within the microstructure. The results show that copper (Cu) is enriched in localized areas within the ferrite matrix, indicating the formation of Cu-rich precipitates. Nickel (Ni) is depleted in the ferrite regions and mainly distributed within the martensitic matrix. Niobium (Nb) forms distinct clusters, suggesting the presence of Nb-rich precipitates or carbides. Oxygen (O) is concentrated around oxide inclusions, while silicon (Si) and manganese (Mn) are sparsely distributed, indicating their presence in small inclusions or at grain boundaries. This is because, at the sintering temperature of 1330 °C, Ni preferentially partitions into the martensitic phase, stabilizing it, while the ferrite remains Ni-depleted. The limited solubility of Cu in the matrix during cooling leads to its precipitation within the ferrite. Nb clusters form due to their tendency to create carbides or precipitates, and oxides develop from reactions between residual oxygen and metallic elements

during sintering. Similar results have been observed in previous studies on sintered 17-4 PH stainless steels [112,191,192].

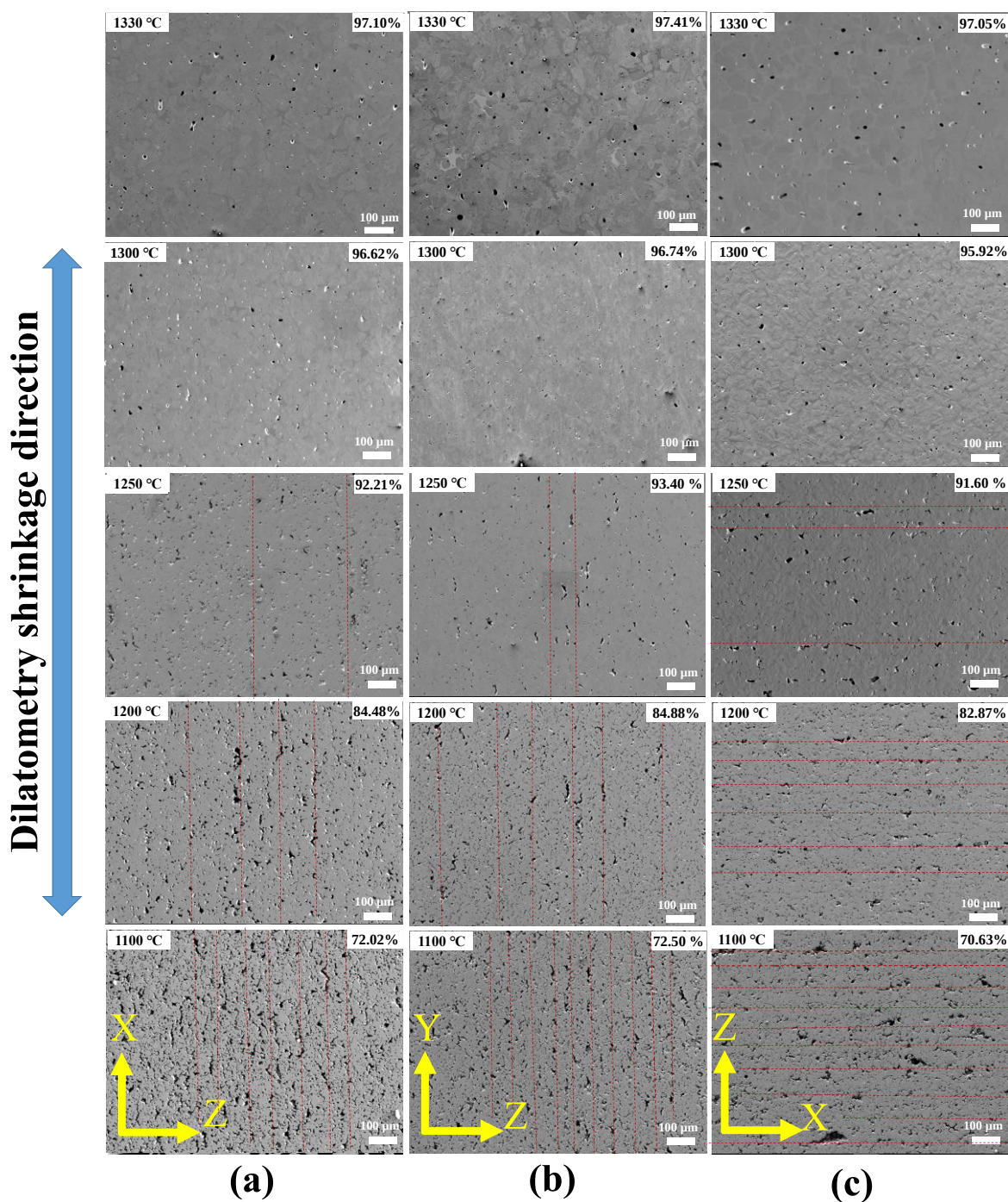


Figure 6-7 Low-magnification SEM images of sintered 17-4 PH stainless steel at five different temperatures ranging from 1100 °C to 1330 °C for three orientations: (a) X-axis, (b) Y-axis, and (c) Z-axis. The part relative density is given in the upper right corner of each figure.

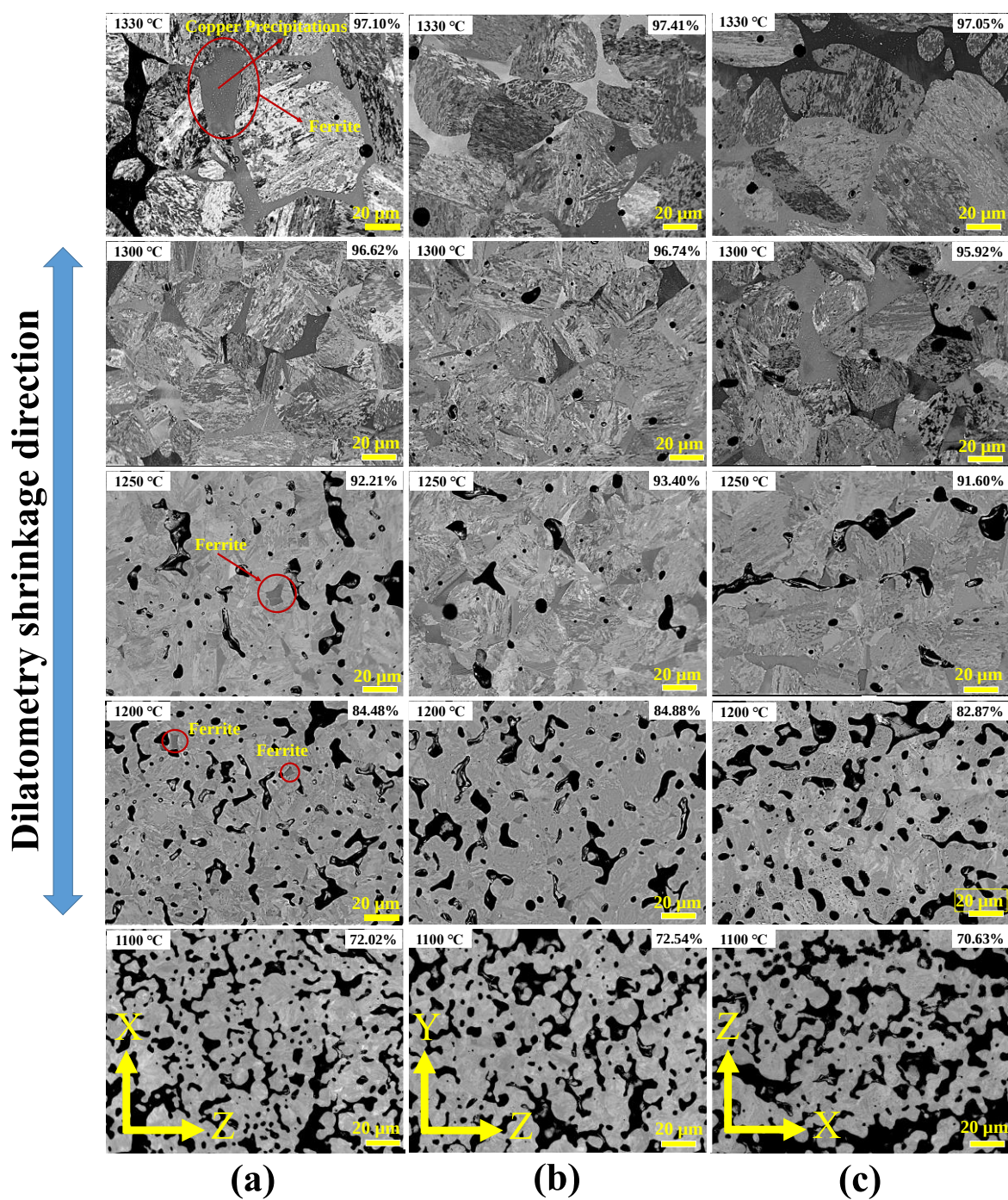


Figure 6-8 SEM images of sintered 17-4 PH stainless steel at five different temperatures ranging from 1100 °C to 1330 °C for three orientations: (a) X-axis, (b) Y-axis, and (c) Z-axis. The part's relative density is given in the upper right corner of each figure.

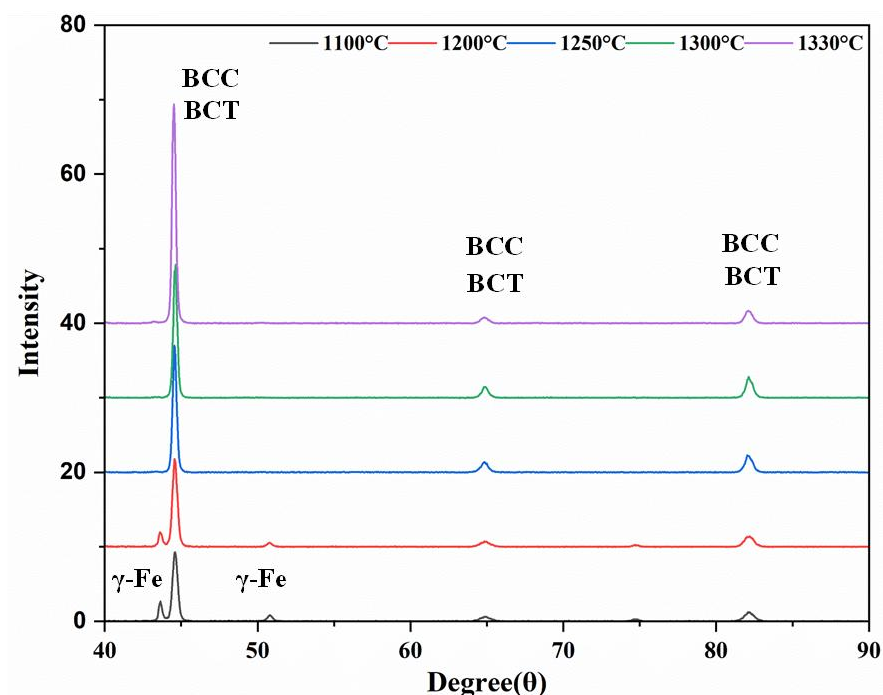


Figure 6-9 X-ray diffraction (XRD) patterns of binder-jetted 17-4 PH samples sintered at various temperatures (1100 °C to 1330 °C). The diffraction peaks correspond to BCC/BCT martensitic phases and residual γ -Fe (austenite).

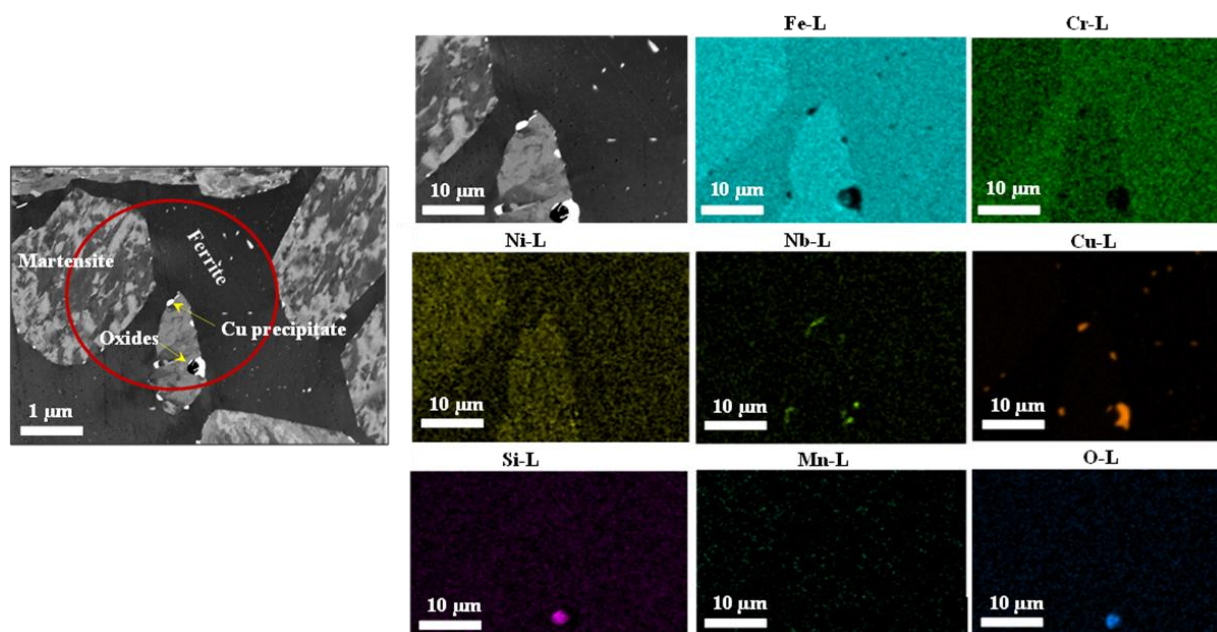


Figure 6-10 The high-magnification SEM image (left) and corresponding EDS elemental maps (right) of BJ SS 17- 4PH sintered at 1330 °C.

6.3.4 Numerical modelling results

Fig. 11 compares the dilatometer strains to the finite element model predictions for the X and Z direction in the powder bed and four sintering temperatures: 1100 °C, 1250 °C, 1300 °C, and 1330 °C. The simulation has three identifiable stages: the elastic expansion, the narrow temperature range where most of the densification happens, and the tail-off till the end of the hold time, which has already been explained in Fig. 5.

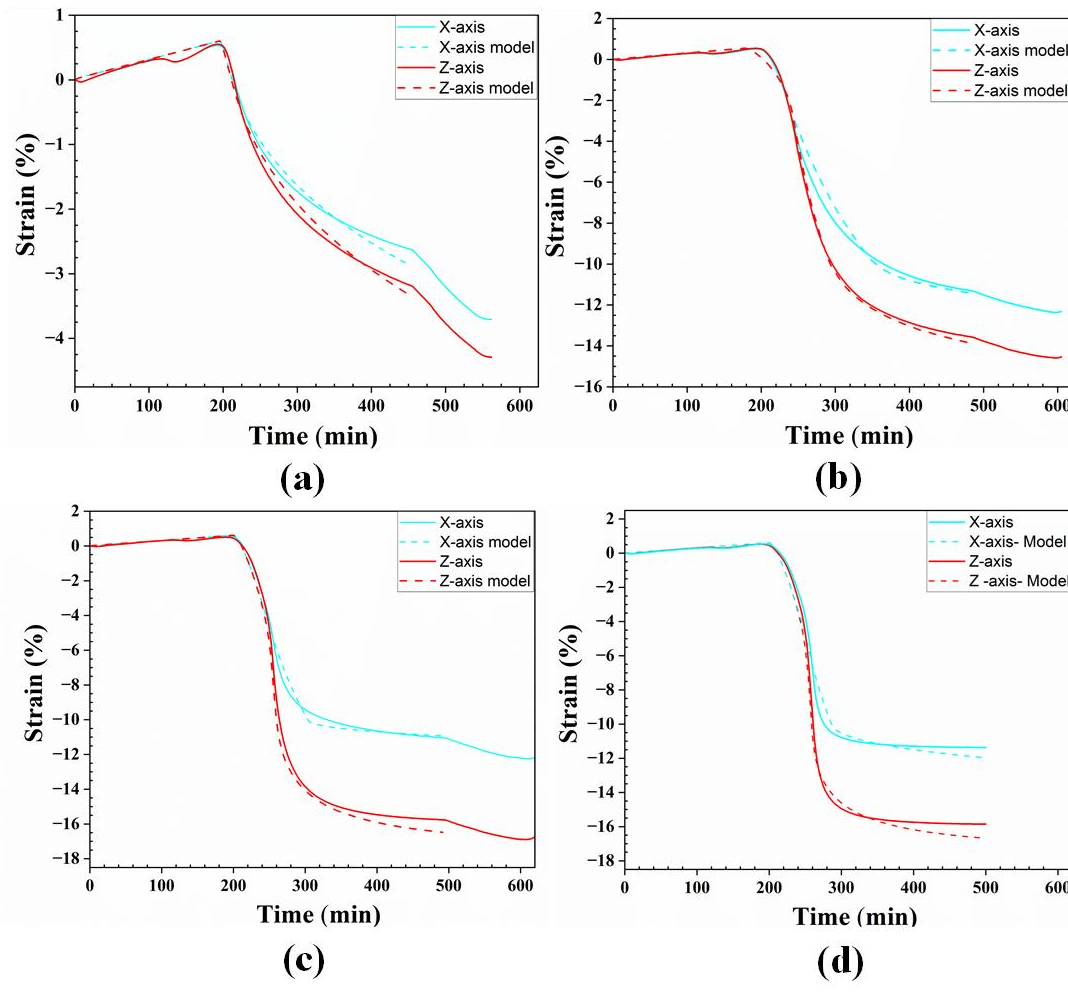


Figure 6-11 Strain vs. time curve for X-axis and Z-axis at multiple sintering temperatures: (a) 1100 °C, (b) 1250 °C, (c) 1300 °C, and (d) 1330 °C. The solid and dashed lines represent the experimental and simulation results, respectively.

The engineering strains from the dilatometer in Fig. 6 are converted into true strains in finite element modeling using equation (23).

$$\varepsilon_{eng} = e^{\varepsilon_{true}} - 1, \quad (23)$$

6.4 Discussion

6.4.1 Effect of sintering temperature on shrinkage rate

Temperature has a profound role in shrinkage, as discussed in Fig. 6. Three different shrinkage regimes (m_1 , m_2 , m_3) are observed with varying temperatures. The strain rate as a function of temperature for the three axes is divided based on the shrinkage regimes in Fig. 12. At lower temperatures (1100–1200 °C), the m_1 regime dominates shrinkage across all printing orientations in Fig. 12 (a) when compared to Fig. 12 (b and c). In Fig. 12 (a), the sintering rate is similar for the X and Y orientations, increasing linearly until 1200 °C to stabilize between -0.09 and -0.10 %/min. The Z-axis shows a more pronounced drop around 1150 °C to reach approximately -0.12 %/min, indicating slight anisotropy in early-stage sintering.

Above 1250 °C, the m_2 regime becomes dominant in Fig. 12 (b), with strain rates increasing approximately four times more than the m_1 regime (-0.16 to -0.50 %/min). The X and Y axes experience a sharp decline from -0.15 %/min at 1300 °C to -0.35 %/min at 1330 °C, while the Z-axis starts at -0.35 %/min and further drops to -0.50 %/min. This pronounced drop for the Z-axis suggests a higher susceptibility to densification-induced shrinkage, reinforcing the anisotropic shrinkage behavior during sintering. The rapid increase in shrinkage rate in the m_2 regime highlights the acceleration of densification at these elevated temperatures. The m_3 regime, corresponding to the isothermal phase, shows a much lower shrinkage rate (-0.005 to -0.001 %/min) (Fig. 12c). This indicates that most shrinkage occurs during the heating phase (m_2 regime above 1250 °C) rather than during the isothermal hold. The same behavior has been previously observed in a different stainless steel [175].

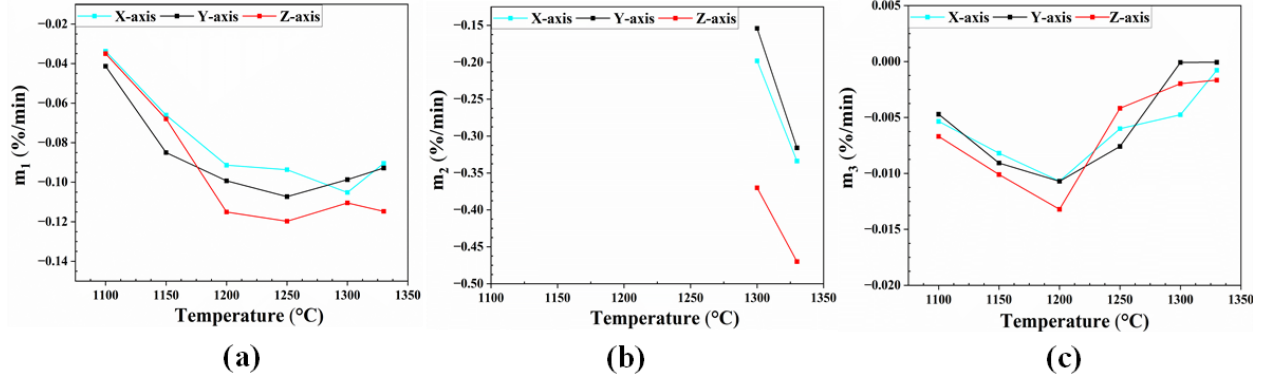


Figure 6-12 Sintering rates at different temperatures for the (a) m_1 , (b) m_2 , and (c) m_3 stages, shown for samples printed in the X, Y, and Z directions.

6.4.2 Activation energy for sintering of binder jet 17- 4PH

The rate of shrinkage varies between the m_1 and m_2 regimes as shown in Fig. 13. It reflects a temperature-induced transition in the dominant sintering mechanism. To determine the active sintering mechanism in each stage, the activation energy for densification can be assessed using the approach proposed by Johnson et al. [154], as presented in equation (24):

$$\ln\left(-T \frac{dL}{dtL_0}\right) = -\frac{Q}{RT} + \ln\left(\frac{F(\rho)AD_0}{G^c}\right) = -n\left(\frac{1}{T}\right) + K, \quad (24)$$

In this equation, dL/L_0 represents the experimentally measured linear shrinkage, which depends on temperature (T). R is the universal gas constant (8.314 J/mol·K), and Q represents the activation energy associated with densification. $F(\rho)$ is a function related to the relative density, and A , c are constants determined by the material's properties. D_0 refers to the pre-exponential factor of the diffusion coefficient, and G indicates the grain size. In Fig. 13, the active sintering mechanism is shown at different sintering temperatures. In this figure $\left(\ln\left(-T \frac{dL}{dtL_0}\right)\right)$ is plotted versus $1/T$ using the 1330 °C dilatometer results extracted from Fig. 5. The slope (n) is utilized to calculate the activation energy (Q) using the relationship ($n = -Q/R$) from equation (24) [151,155–158]. Fig. 13 presents the measured slopes and the corresponding activation energies for the m_1 and m_2 regimes. Note that the activation energies for different printing orientations calculated from the dilatometric curves at various temperatures in Fig. 6 are nearly identical.

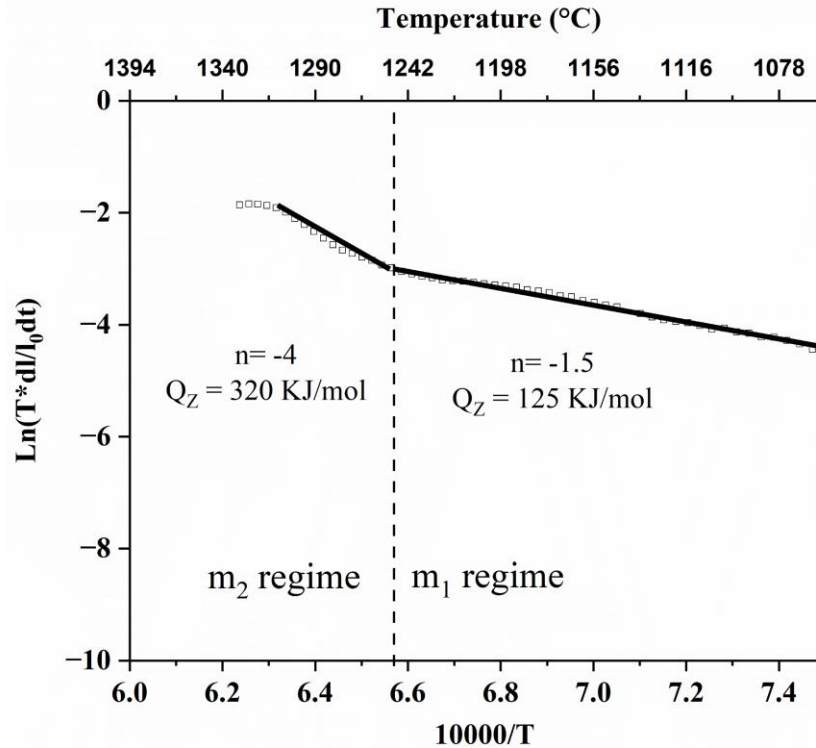


Figure 6-13 Activation energy (Q) as a function of temperature across distinct sintering shrinkage regimes for the Z-axis printed sample sintered up to 1330 °C.

Sintering between 1000 °C and 1250 °C corresponds to low activation energies (100–125 kJ/mol), indicating the dominance of grain boundary diffusion [159,193]. This mechanism is more significant at low and intermediate temperatures because of the higher availability of vacancies and the increased presence of the austenite grain boundaries, acting as pathways for enhanced atomic mobility [151]. At lower temperatures within the m_1 regime, the structure comprises interconnected voids, retained austenite, and martensite (Fig. 9). Fig. 14 shows the evolution of grain size (Fig. 14 (a)) and density (Fig. 14 (b)) with temperature. During the m_1 regime (between 1100 and 1200°C), changes in grain size (10–12 μm) and final density remain minimal [194,195]. This is because pores act as obstacles to grain boundary motion, exerting Zener drag that suppresses grain growth [160]. Consequently, densification is restricted, resulting in limited shrinkage ($\sim 4\%$, as shown in Fig. 6) and low sintered densities ($<80\%$, as shown in Fig. 14b).

Interestingly, XRD results in Fig. 9 show evidence of retained austenite for sintering temperatures between 1100 °C and 1200 °C. This is uncommon for 17-4 PH since the austenite-martensite transition temperature occurs around 130 °C [196,197]. A similar observation has been reported

when 17-4PH is processed by MIM technology [177]. This phenomenon can be attributed to carbon from the residue binder, which acts as an austenite stabilizer, delaying the transformation of austenite to martensite during cooling. Above 1200 °C sintering temperature, the retained austenite peak disappears (Fig. 9), which suggests that increasing sintering temperature allows burning of more carbon from the material. At temperatures above 1200 °C, shrinkage is governed by the m_2 regime, which is characterized by higher activation energies (250 – 320 kJ/mol), as shown in Fig. 13. This regime indicates that mass transport and densification are predominantly controlled by volume diffusion [110,115,193,198]. Volume diffusion is based on atomic movement through the crystal lattice, which becomes more efficient at higher temperatures due to increased atomic mobility. Since there are fewer obstacles within the grain interiors, this mechanism leads to greater shrinkage, as observed in Fig. 14(b). Under this regime, grain size increases significantly from 10 to 80 μm (Fig. 14(a)) with the reduction of Zener drag forces [199]. Fig. 14(c) shows the effect of temperature on δ -ferrite formation. At 1200 °C, very few δ -ferrites (2 %) can be observed. Above 1250 °C, the sharp increase in density observed in Fig. 14(b) coincides with the increase in δ -ferrite volume fraction in Fig. 14(c). Increasing δ -ferrite from 7 % to 22 % is associated with density increases from 83 % to 97 %.

Microstructures (Fig. 8) show that δ -ferrite forms close to existing pores, accelerating the diffusion rate and promoting the transformation from elongated pores below 1250 °C to more spherical pores above 1250 °C. The effect of phase transformation on the sintering kinetics of binder jet parts is similar to that of conventional powder metallurgy. However, the effect of carbon residue from the binder is not negligible and affects the phase transformation temperatures and, thus, sintering kinetics. The transition temperature between grain boundary and bulk diffusion, 1250 °C, observed here is slightly higher than what is normally reported in the literature (1000–1150 °C) [177]. Similar observations have been reported for BJ 316L [175]. The debinding step before sintering is thus a crucial operation that must not be overlooked to optimize the operation.

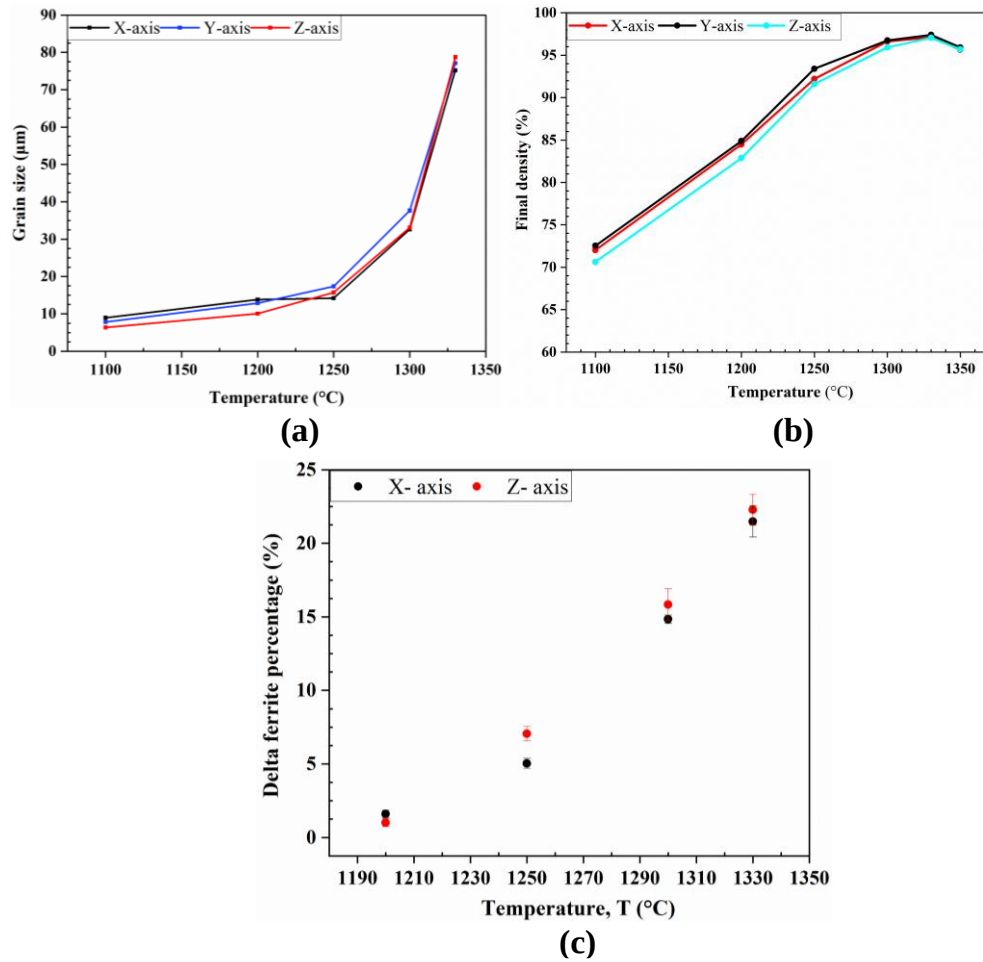


Figure 6-14 Influence of sintering temperature on (a) grain size, (b) final density, and (c) δ -ferrite percentage in binder-jet 17-4 PH samples

6.4.3 Anisotropic shrinkage

Fig. 15 illustrates the shrinkage behavior and anisotropy during the sintering of BJ stainless steel 17-4 PH. The Fig. 15(a) quantifies shrinkage along three orthogonal directions (ϵ_X – red, ϵ_Y – black, and ϵ_Z – blue), while the Fig. 15(b) focuses on the anisotropic shrinkage ratio (ASR) to highlight differences in shrinkage behavior across these directions. Shrinkage percentages show a consistent increase with temperature from 1100 $^{\circ}\text{C}$ to 1350 $^{\circ}\text{C}$. At 1100 $^{\circ}\text{C}$, the shrinkage values start around 3-4 % for all three directions, indicating minimal densification at lower temperatures. As the temperature increases, the shrinkage becomes more pronounced, particularly beyond 1200 $^{\circ}\text{C}$. By 1330 $^{\circ}\text{C}$, the Z-direction exhibits the highest shrinkage, reaching approximately 18 %, while the X- and Y-directions exhibit lower shrinkages of approximately 13 % and 14 %, respectively. These results highlight anisotropic behavior, where the Z-direction undergoes significantly more

shrinkage, about 30 %-40 % higher than the X-Y plane. Fig. 15(b) quantitatively captures the anisotropy using the anisotropic shrinkage ratio (ASR). The ratio $\epsilon_z/(\epsilon_x + \epsilon_y)/2$, shown by the red curve, increases steadily with temperature, starting near 1.0 at 1100 °C and reaching approximately 1.4 at 1330 °C. This indicates that by 1330 °C, the shrinkage in the Z-direction is 40 % greater than the average shrinkage in the X-Y plane. This rising trend confirms that anisotropy becomes more pronounced as sintering progresses. On the other hand, the ratio ϵ_y/ϵ_x (black line) remains close to 1.05 throughout the temperature range, signifying that shrinkage within the X-Y plane remains quasi-isotropic. The fitted polynomial equation (Table 4) provides a robust model for ASR in the Z-axis, with a high R-squared value (0.98), confirming the reliability of the trend. The increasing ASR emphasizes that as sintering progresses, anisotropy becomes more pronounced, primarily affecting the Z-direction. The shrinkage and anisotropy behavior during the sintering of 17-4 PH stainless steel, as seen in Fig. 15, is strongly influenced by the presence of primitive lines in the build direction (Z-direction). These primitive lines formed during the layer-by-layer fabrication process, leading to greater shrinkage and pronounced anisotropy at intermediate sintering temperatures such as 1200 °C and 1300 °C. At 1200 °C, the shrinkage in the Z-direction (ϵ_z) is significantly higher (~10 %) compared to the X-Y plane (~7.5 %-8 %), resulting in an ASR of 1.25. This is because the primitive lines along the Z-direction are still present and undergo substantial densification and collapse during sintering. Similarly, at 1300 °C, the Z-direction shrinkage increases further to ~16 %, while the X-Y shrinkage rises more moderately to ~12 %-13 %. The ASR reaches 1.35 at this stage, emphasizing that the Z-direction continues to densify more due to the elimination of remaining primitive lines. The differential shrinkage highlights the anisotropic behavior caused by the structural characteristics of the build direction. At higher sintering temperatures, such as 1330 °C, the influence of primitive lines diminishes as they have largely been eliminated during the earlier stages of sintering. Consequently, the anisotropy in shrinkage begins to stabilize, as indicated by the near-constant ASR of ~1.4. The absence of primitive lines means that the remaining densification mechanisms, such as grain boundary and bulk diffusion, act more uniformly across all directions. This results in a more isotropic behavior, where further shrinkage differences between the Z-direction and the X-Y plane become less pronounced.

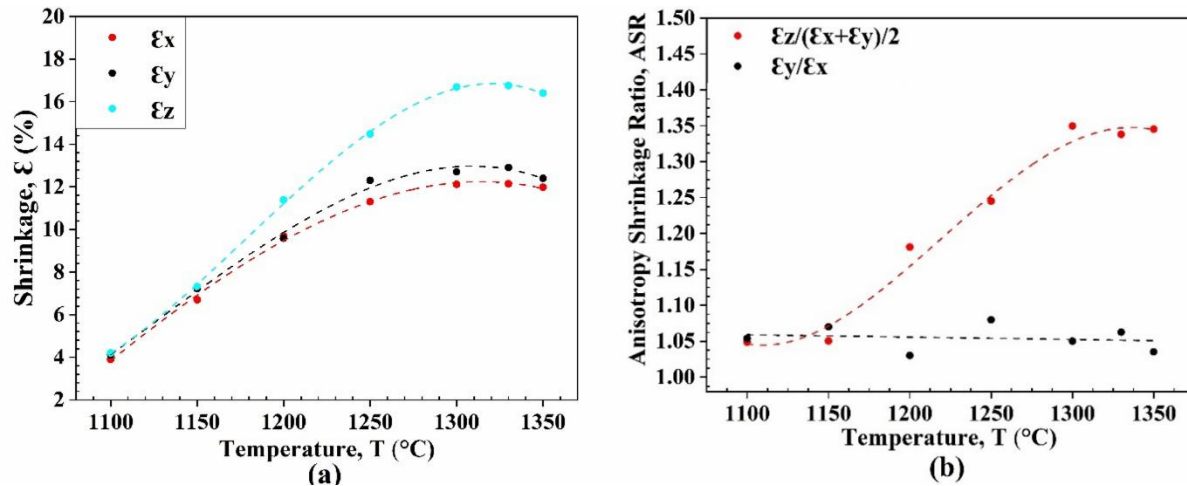


Figure 6-15 (a) Shrinkage strain as a function of sintering temperature for different print orientations: ϵ_x – red, ϵ_y – black, and ϵ_z – blue; (b) Temperature-dependent anisotropic shrinkage ratios (ASR), including in-plane (ϵ_y / ϵ_x) and out-of-plane ($\epsilon_z/((\epsilon_x + \epsilon_y)/2)$).

Table 6.4 Fitted equations and corresponding R^2 values for the anisotropic shrinkage data presented in Fig. 15(b).

	$\epsilon_z/(\epsilon_x + \epsilon_y)/2$
Fitted Equation	$\text{ASR} = 2.23 * E - 11 X^4 - 1.66 * E - 7X^3 + 3.99 * E - 4X^2 - 0.398 * X + 143$
R-Squared Value	0.98

6.4.4 Effect of anisotropic shrinkage on activation energy

Fig. 16 highlights two distinct kinetic regimes (m_1 and m_2), each defined by different activation energies. In the high-temperature regime (m_2), the activation energy is 320 kJ/mol across all three axes, while in the low-temperature regime (m_1), it decreases to 125 kJ/mol. Despite these variations, the shrinkage behavior along the X, Y, and Z axes remains nearly identical, demonstrating isotropic activation energy behavior during the sintering process. This is not surprising since the activation energy for sintering is mostly dependent on chemical composition. Uniformity in activation energy contributes to similar microstructural evolution during sintering, including grain sizes (Fig. 14(a)), ferrite fraction (Fig. 14(c)), and bulk densities (Fig. 14(b)) for the X-, Y-, and Z-axes.

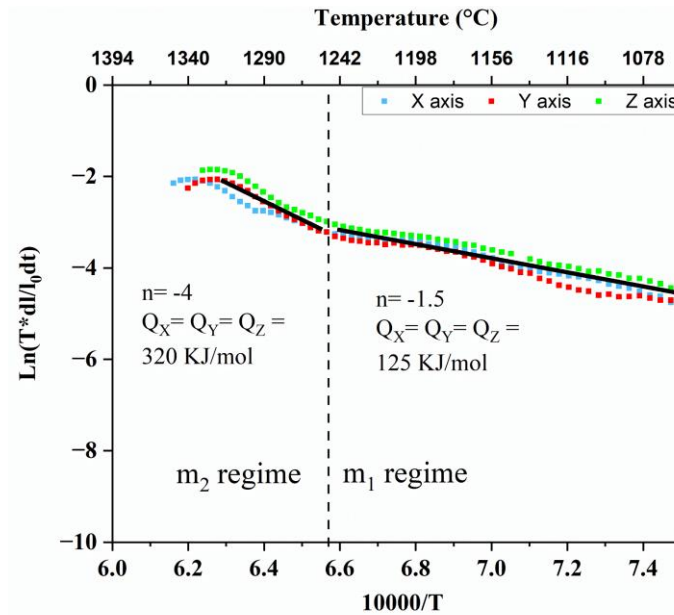


Figure 6-16 Effect of temperature on activation energy (Q) across the m_1 and m_2 sintering shrinkage regimes for samples printed along the X, Y, and Z axes and sintered up to 1330 °C.

6.4.5 Effect of anisotropic pore distribution on viscous moduli

During sintering, the mechanical responses to volumetric compression (resulting from shrinkage and pore collapse) and shear deformation (caused by uneven stress distribution or anisotropic behavior) are controlled by the bulk viscosity modulus (K) and shear viscosity modulus (G) [200]. These two moduli are material properties that depend on the microstructure and vary with pore density and distribution. Since these moduli can hardly be obtained experimentally, the two moduli can be extracted from the sintering model described in section 2.5. Fig. 17 shows the variation of G (extracted from equation (18)) and K (extracted from equation (16)) as a function of porosity (θ) in two printing orientations, X-axis and Z-axis, during sintering at different temperatures: (a) 1100 °C, (b) 1200 °C, (c) 1250 °C, and (d) 1300 °C and (e) 1330 °C. G and K increase as porosity (θ) decreases at all sintering temperatures. This is because denser materials, with better particle bonding and reduced pore volume, exhibit greater resistance to shear deformation and volumetric compression [81,201–203].

The effect of sintering temperature on these viscous moduli is further highlighted in **Fig. 18**, which shows the temperature dependence of G and K for samples printed along the X-axis. With increasing sintering temperature, the shear modulus decreases steadily. This decline is primarily attributed to the reduction in viscosity at elevated temperatures. During the early to intermediate

stages of sintering, increased temperatures enhance atomic diffusion, facilitating neck growth and particle rearrangement [151]. However, at higher temperatures, the material softens, leading to a diminished ability to resist shear deformation [204]. Furthermore, K shows similar behavior except for the region near the sintering temperature of 1250 °C (T_{critv2}), where a transition in grain growth kinetics occurs. This leads to a sudden increase in material density (Fig. 14(b)), causing a large impact on the bulk modules. However, at higher temperatures, the effect of temperature becomes dominant. As porosity plays a lesser role after 1250 °C, thermal and microstructural degradation lead to a decline in bulk modulus. This is consistent with previous studies [61,200,205].

The results in Fig. 17 demonstrate that material densification during the sintering process exhibits anisotropic behavior. Both simulation and experimental findings indicate that deformation in the Z direction is more pronounced than in the X direction, an outcome attributed to anisotropic viscosity behavior that results in higher viscosity along the Z axis. These findings are consistent with previous studies by Jamalkhani et al. [144]. At higher porosity levels, the overall material density is strongly influenced by the shape of the pores and their directional distribution. On the other hand, at higher sintering temperatures, shrinkage in pore size leads to a more homogenous pore distribution and similar G and K values in the X and Z directions.

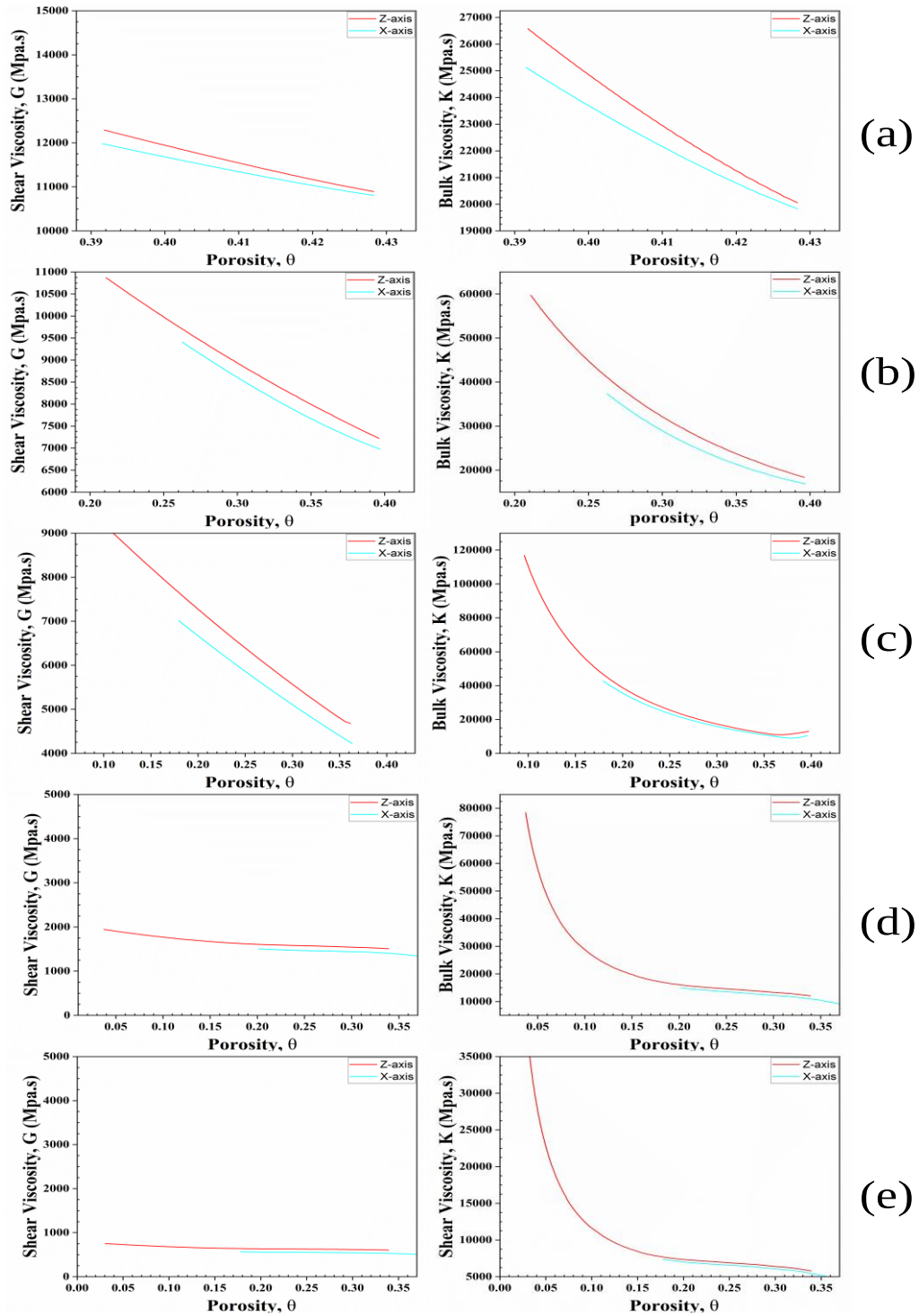


Figure 6-17 Bulk and shear viscosity during the sintering at: (a) 1000 °C, (b) 1200 °C, (c) 1250 °C, (d) 1300 °C, and (e) 1330 °C.

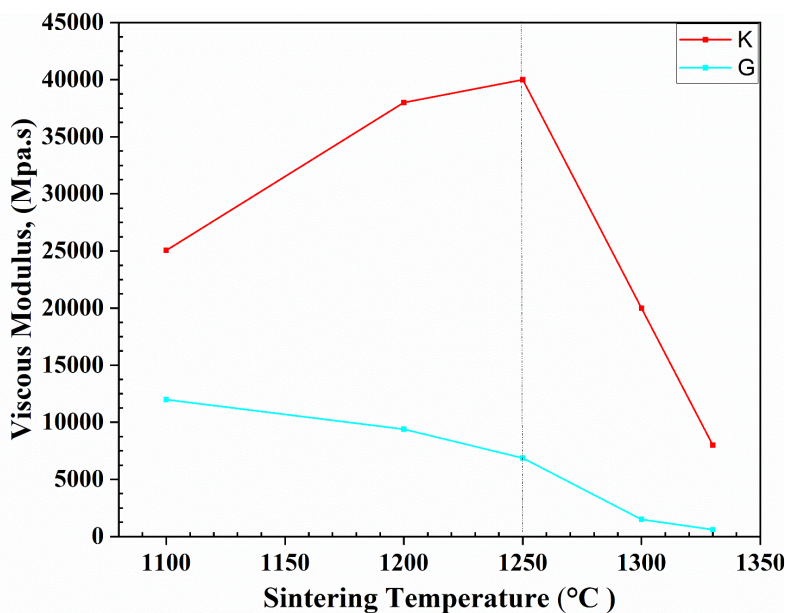


Figure 6-18 Variation of bulk viscosity modulus (K) and shear viscosity modulus (G) as a function of sintering temperature for samples printed in the X axis.

Based on the combined experimental and modeling results, several interconnected mechanisms can be identified that govern the anisotropic sintering behavior. The collapse of primitive lines aligned perpendicular to the build direction (Z-axis) plays a primary role in promoting directional shrinkage. These features, inherent to the layer-by-layer binder jetting process, act as structural weaknesses that densify preferentially at elevated temperatures (Fig. 7, Fig. 15). Above 1250 °C, the dominant diffusion mechanism shifts from grain boundary to volume diffusion, enhancing atomic transport and promoting rapid grain growth (Fig. 13). The formation of δ -ferrite near residual pores further accelerates local densification, particularly in regions influenced by primitive line collapse (Fig. 8). In addition, the anisotropic pore morphology in the green part leads to higher shear and bulk viscosity moduli in the Z-direction, reinforcing directional deformation (Fig. 17). As sintering progresses and pores become more uniformly distributed, the anisotropy in both structure and shrinkage response gradually diminishes (Fig. 7). These mechanisms explain the temperature- and orientation-dependent evolution of anisotropic shrinkage in binder-jetted 17-4 PH stainless steel.

6.5 Conclusions

The anisotropic sintering behavior in binder-jetted 17-4 PH stainless steel was investigated through an integrated experimental and modeling framework. Six different sintering temperatures and three

printing orientations were examined. The combined approach provided a predictive tool to understand and control anisotropic shrinkage during sintering, supporting improved dimensional accuracy and processing strategies for binder-jetted 17-4 PH components. The key conclusions drawn from this study are as follows:

- 1- An anisotropic shrinkage ratio (ASR) of up to 1.4 is observed in the Z-direction of binder-jetted 17-4 PH stainless steel, indicating up to 40 % higher shrinkage compared to the X–Y plane where quasi-isotropic shrinkage occurs. This behavior becomes more pronounced at temperatures above 1250 °C, primarily due to the collapse of primitive lines perpendicular to the build direction.
- 2- All printing orientations exhibit comparable microstructural evolution, characterized by grain growth from 10 to 80 μm , an increase in δ -ferrite content from 2 % to 22 %, and final densities up to 97 % at 1330 °C. This uniform behavior results in similar activation energies and a consistent transition temperature across all directions.
- 3- Dilatometry experiments and viscoplastic modeling identified a sintering mechanism transition around 1250 °C, where the dominant diffusion mode shifts from grain boundary diffusion ($Q = 100\text{--}125 \text{ kJ/mol}$) to volume diffusion ($Q = 250\text{--}320 \text{ kJ/mol}$), with similar activation energies along all axes. This transition temperature is slightly higher than that in conventional powder metallurgy, likely due to carbon residue from the binder.
- 4- The temperature dependence of the shear (G) and bulk (K) viscosity moduli highlights a critical transition near 1250 °C. Below this temperature, G decreases gradually due to material softening, while K shows a sharp increase linked to rapid densification and grain growth. Above 1250 °C, both moduli decline as thermal effects and microstructural degradation reduce the material's resistance to deformation.
- 5- Finite element simulations and experimental results confirm that sintering behavior in binder-jetted 17-4 PH stainless steel is initially anisotropic due to directional pore distribution, leading to higher shear ($G = 12000 \text{ MPa}\cdot\text{s}$) and bulk ($K = 26400 \text{ MPa}\cdot\text{s}$) viscosity moduli along the Z-axis. However, as sintering temperature increases and porosity decreases, pores become more uniformly distributed, promoting isotropic shrinkage and resulting in similar shear and bulk modulus across all directions.

Acknowledgments

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CHAPTER 7 ARTICLE 3: Effect of powder size distribution on anisotropic shrinkage of Binder Jet 316L stainless steel parts

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Abstract

Powder size distribution (PSD) strongly influences green density, densification, and dimensional stability during sintering in binder jet additive manufacturing (BJAM). This study employed dilatometry to analyze the dimensional evolution of cubic 316L stainless steel samples fabricated by binder jetting, focusing on the influence of PSD on anisotropy and microstructural development. Two powders were investigated under identical conditions: MIM-cut with a narrow PSD and LPBF-cut with a broader PSD. MIM-cut samples exhibited higher linear shrinkage in the Z- and X-directions (17.8% and 12.6%, respectively) compared to LPBF-cut samples (15% and 12.4%, respectively) at the maximum sintering temperature. The anisotropy shrinkage ratio (ASR) exceeded 1.4 for MIM-cut samples, while LPBF-cut samples showed a lower ASR (~1.2) due to their broader PSD, which promoted higher green density and more isotropic shrinkage. Despite their lower initial green density, MIM-cut specimens achieved a higher final relative density (~97 %) than LPBF-cut specimens (~95 %), owing to enhanced densification kinetics provided by their finer, narrower PSD. These findings highlight the critical role of PSD in governing densification, dimensional accuracy, and microstructural development in binder jet additive manufacturing.

Keywords: Additive manufacturing, anisotropic sintering shrinkage, particle size distribution, stainless steel

7.1 Introduction

In recent years, additive manufacturing (AM) has emerged as a transformative technology, with applications ranging from prototyping to full-scale industrial production [132,206]. This has led to the development of several powder-based AM processes being increasingly applied to a wide range of materials [170,207,208]. Binder jet additive manufacturing (BJAM), one of the prominent powder-based AM techniques, has gained significant attention, offering advantages such as high build speed, low residual stress, and compatibility with a wide range of alloys [79,209]. Among these, austenitic stainless steel 316L is widely used due to its excellent corrosion resistance, ductility, and processability in both as-sintered and post-processed states [13,87,130,151,175,210,211]. However, a key challenge in BJAM remains controlling shrinkage, densification, and microstructural instability during sintering, which are crucial for achieving dimensional accuracy and obtaining the desired mechanical properties [14,15,142,212,213].

Powder size distribution is a key factor governing sintering kinetics, green part density, and the overall quality of binder jet additively manufactured (BJAM) components [46,214]. Powders with a narrow PSD generally result in lower green densities because of limited packing efficiency. In contrast, broader PSDs allow finer particles to fill the interstitial spaces between coarser ones, thereby improving green density [53,215]. However, despite the lower green density, powders with a finer and narrower PSD often achieve higher sintered densities due to their smaller pore sizes and increased surface area, which enhances the thermodynamic driving force for densification [68,210,216].

German [151] and Rahaman [217] investigated the effect of PSD on sintering properties in the metal injection molding process, emphasizing the direct effect of PSD on pore structure, shrinkage, and grain growth during sintering. Mostafaei et al. [218] also investigated the effect of PSD on the BJ-IN625, demonstrating finer powders with a narrower PSD enhanced densification, but also leads to increased anisotropic shrinkage during super-solidus liquid phase sintering due to uneven sintering rates along different build directions.

Although the influence of PSD on sintering has been studied in stainless steels and other alloys, a clear understanding of how PSD alone affects anisotropic shrinkage, densification, and microstructural evolution in binder-jetted 316L stainless steel is still lacking. In particular, the impact of PSD on phase development, especially δ -ferrite formation, has not been systematically

addressed. Therefore, a comprehensive investigation is required to elucidate the effects of powder size distribution on shrinkage behavior, anisotropy, microstructural evolution, and phase transformation in binder-jetted 316L stainless steel.

This study addresses the gaps in available literature by systematically comparing two powders with distinct PSD characteristics: MIM-cut powder with a narrow distribution and LPBF-cut powder with a broader distribution, both processed under identical binder jetting and sintering conditions. Through a combination of dilatometry and microstructural analysis, the influence of PSD on densification kinetics and sintering shrinkage is quantified, revealing anisotropic shrinkage. The results provide critical insights for optimizing powder selection and sintering strategies in binder jet additive manufacturing.

7.2 Experimental procedures

7.2.1 Materials

Gas-atomized austenitic stainless steel 316L powder of two different grades: (i) MIM-cut with a narrower PSD and (ii) LPBF-cut with a broader PSD, has been used in this study. The particle size distribution for both the powders shown in Figure 1 was obtained using the CAMSIZER X2 system (RETSCH, Wuppertal, Germany). MIM-cut powder demonstrated a narrower PSD (Figure 1a), while the LPBF-cut powder exhibited a broader distribution (Figure 1b) due to a wider range of particle sizes. The nominal chemical composition is provided in Table 1.

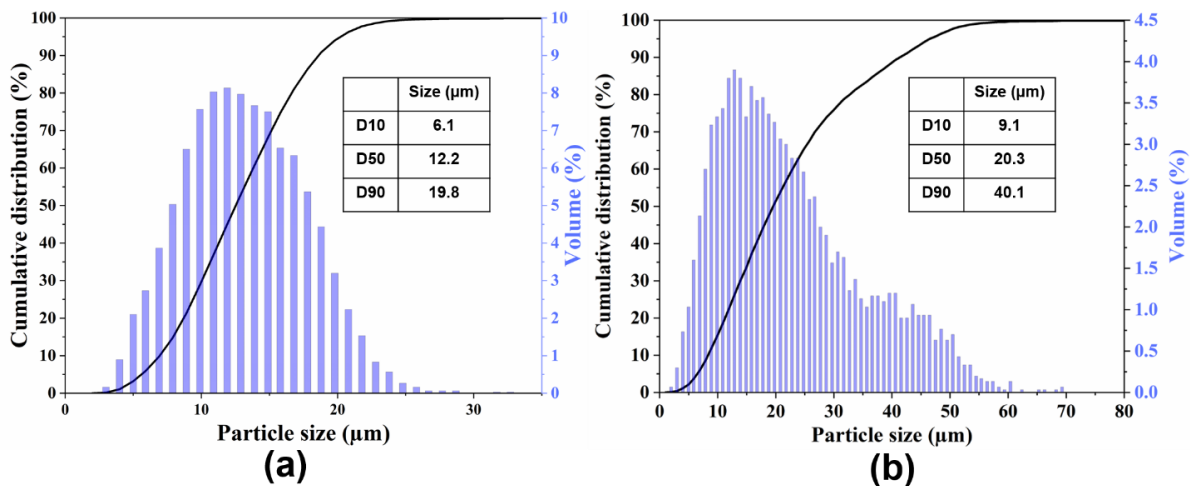


Figure 7-1 Particle size distribution of (a) MIM-cut powder and (b) LPBF-cut powder of BJ 316L.

Table 7.1 Chemical composition of gas atomized SS 316L powder (wt.% %).

Cr	Ni	Mo	Mn	Si	C	O	S	Fe
16.0-18.0	10.0-14.0	2-3	≤2.0	≤1.0	≤0.03	≤0.14	≤0.04	Bal.

7.2.2 BJ Printing and Characterization

Cuboidal test specimens in the dimensions of 10 mm × 10 mm × 20 mm were printed in X- and Z-direction using a commercial ExOne Innovent+ binder jet printer. Specimens were printed in both directions to assess the impact of build orientation on shrinkage behavior. The LPBF-cut samples exhibited green densities of 58–60%, whereas the MIM-cut samples showed slightly lower values of 56–57%.

Post printing, green parts were thermally cured at 180 °C for 6 hours to evaporate residual binder solvent. Thermal debinding was performed in an Ar atmosphere at 600 °C for 4 hours to remove organic binder residues. Sintering was conducted under a 95% Ar–5 % H₂ reducing atmosphere in a horizontal tube furnace. The sintering cycle comprised heating at a rate of 5 °C/min to target temperatures between 1100 and 1390 °C, followed by a 6-hour isothermal hold at the peak temperature. The specimens were then cooled to room temperature at a rate of 5 °C/min in the same reducing atmosphere. The experimental setup and representative printed specimens are shown in Figure 2. Shrinkage behavior was continuously monitored using a horizontal push-rod dilatometer (Netzsch DIL 402S). The final dimensions of the sintered specimens were recorded using a precision caliper to validate the dilatometric data. The sintered density was determined by Archimedes' method in accordance with ASTM B962-13 [219].

Metallography specimens were prepared using standard procedure, grinding with abrasive paper successively from (mention starting and end grade of paper), followed by polishing using diamond suspension and 0.04 µm colloidal silica. The polished samples were then ultrasonically cleaned in distilled water for 5 minutes. Optical microscopy was performed using a Keyence VHX-7000 digital microscope to evaluate porosity and quantify grain size in green and sintered parts. Further examination of microstructural features, including porosity morphology and grain characteristics, was carried out using a JEOL JSM-7600F field-emission scanning electron microscope (FEG-SEM) equipped with an Oxford Instruments X-Max 80 mm² EDS detector for elemental analysis.

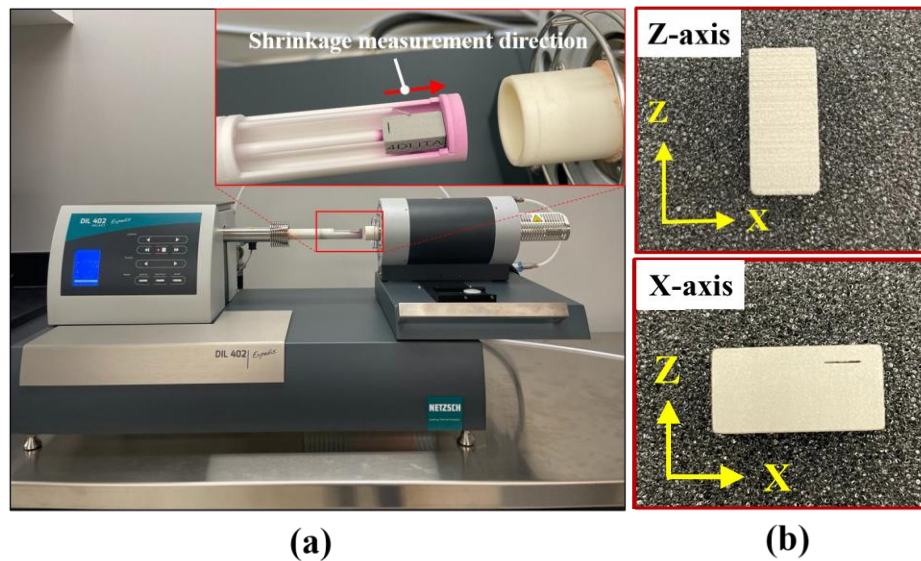


Figure 7-2 (a) Dilatometry sintering setup (b) Samples printed along the Z-axis and X-axis

7.3 Results and Discussion

7.3.1 Effect of PSD on the shrinkage behavior and microstructure

Figure 3 shows the time-dependent linear shrinkage (%) of binder-jetted 316L samples made from MIM-cut and LPBF-cut powders, measured along the X- and Z-directions over the sintering temperature range of 1100 –1390 °C. For MIM-cut powders, shrinkage increases steadily with temperature, reaching approximately 12.6% in the X-direction and 17.8% in the Z-direction at the maximum sintering temperature of 1370 °C. In contrast, LPBF-cut powders sintered up to 1390 °C exhibit lower overall shrinkage, with final values of approximately 12.5% in the X-direction and 15% in the Z-direction. Z-oriented samples exhibit greater shrinkage in both powder types than those oriented along the X-direction, highlighting anisotropic sintering behavior. Anisotropic sintering primarily arises from the nature of the binder jetting process, where the powder is deposited layer by layer along the Z-axis. During printing, the interaction between high-velocity binder droplets and the powder bed induces periodic structural features, referred to as primitive lines, which are perpendicular to the build direction [136,153]. These lines represent regions of localized porosity and weaker interlayer bonding, acting as planes of structural discontinuity that collapse more extensively during the sintering process. As a result, greater shrinkage is observed in the Z-direction compared to the X-direction. Compared to LPBF-cut powders, MIM-cut powders exhibit more pronounced shrinkage at lower temperatures, indicating more efficient densification

behavior. This is primarily attributed to their finer particle size and narrower particle size distribution, which enhance surface diffusion and sintering kinetics despite their relatively lower green density [68]. Similar anisotropic shrinkage behavior has been reported in previous studies by Rahaman et al. [64], Esmati et al. [175,220,221], and Rios et al. [13].

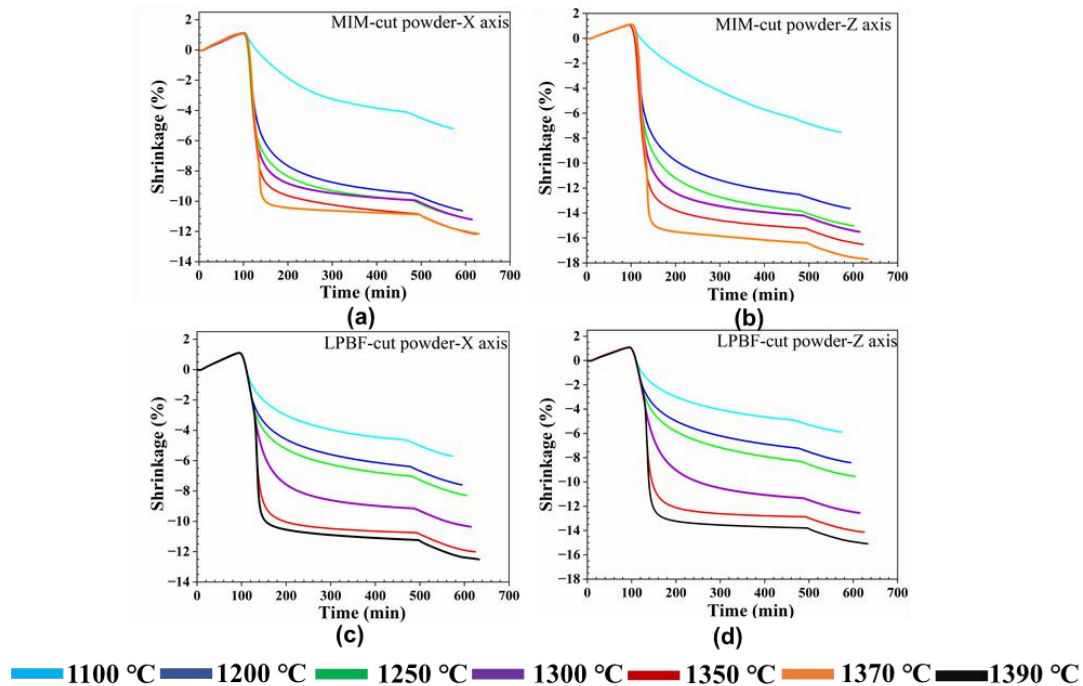


Figure 7-3 Shrinkage curves of binder-jetted 316L samples made from MIM-cut (a, b) and LPBF-cut (c, d) powders along X and Z directions, respectively, at different sintering temperatures.

Figure 4 illustrates the microstructural evolution of binder-jetted 316L samples sintered at different temperatures and orientations for the two PSD conditions. The red arrows highlight regions corresponding to delta ferrite, a high-temperature phase that can form during sintering, particularly at elevated temperatures, as also reported by Rios et al. [13]. The yellow arrows indicate annealing twin boundaries, typical features of recrystallized austenitic stainless steel formed during grain growth. As the sintering temperature increases, the micrographs reveal a clear reduction in porosity and a progressive increase in grain growth. At 1100 °C, all samples exhibit porous structures with limited neck formation. LPBF-cut samples initially show slightly higher relative green densities (~58%) compared to MIM-cut samples (~56%), suggesting better green packing efficiency. However, as sintering proceeds to 1300 °C and 1390 °C, MIM-cut powders promote more rapid densification due to their finer particle size and narrower PSD, resulting in refined and more

equiaxed grain structures. The higher surface area of MIM-cut powders accelerates sintering kinetics and limits the time available for grain coarsening.

Additionally, the abundance of initial particle boundaries may act as grain boundary pinning sites, further restricting grain growth. In contrast, LPBF-cut powders with a broader PSD show slower densification and more pronounced grain growth, leading to coarser and less uniform grains [222,223]. This behavior is consistent with equation 1 [151]:

$$\frac{df}{dt} = K_3 \frac{(1-f)^K}{G^m} \quad (1)$$

Where the densification rate ($\frac{df}{dt}$) is inversely related to grain size (G) and directly related to fractional density (f). German et al. [151] noted that finer powders enhance sintering kinetics by increasing surface area and facilitating early-stage neck formation. This faster densification reduces the thermal exposure needed to reach full density and helps suppress grain coarsening.

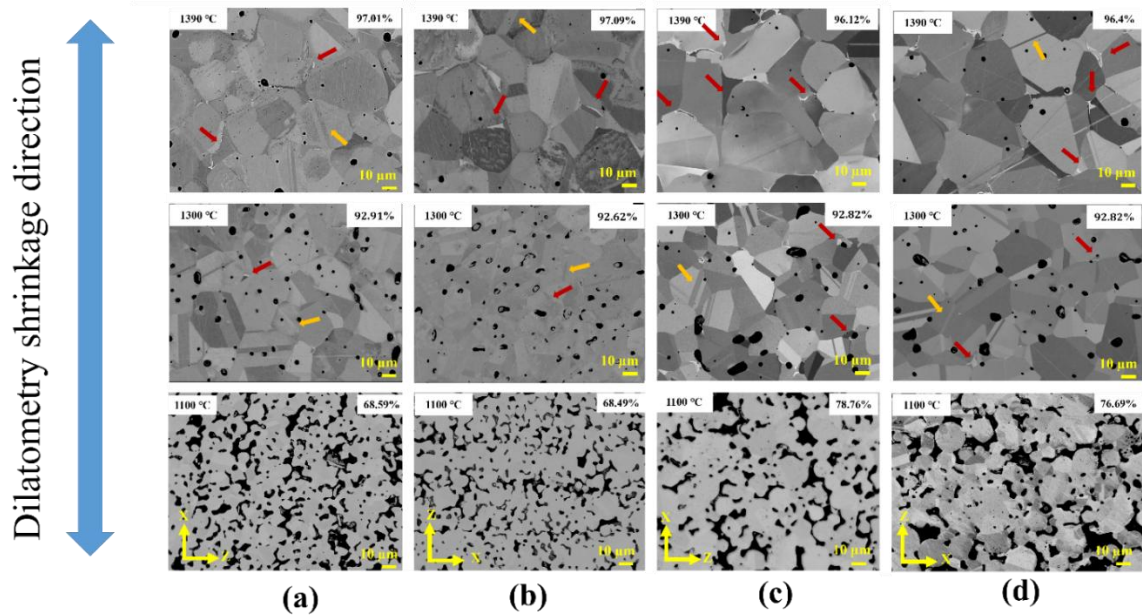


Figure 7-4 SEM images showing the microstructure evolution in binder-jetted 316L samples sintered at 1100 °C, 1300 °C, and 1390 °C for MIM-cut (a, b) and LPBF-cut (c, d) powders along X and Z printing direction.

Quantitative data from Figure 4 show that the MIM-cut powder, sintered at 1370 °C, achieved a relative density of ~ 97%, an average grain size of ~ 46 μm , and a δ -ferrite fraction below 3%. In contrast, the LPBF-cut powder, sintered at 1390 °C, reached a slightly lower density (~ 95–96 %), with a coarser grain size (~ 50 μm) and nearly twice the δ -ferrite content (~ 6 %). The superior densification of MIM-cut powders arises from their finer and narrower PSD, which increases surface area and reduces pore size, thereby enhancing the capillary driving force for sintering [151]. This accelerates neck formation and pore elimination, allowing MIM-cut samples to achieve high density at lower temperatures.

7.3.2 Anisotropic sintering shrinkage

The plots in Figure 5 illustrate the evolution of shrinkage strain and anisotropy in binder-jetted 316L stainless steel samples fabricated from MIM-cut and LPBF-cut powders sintered across a range of temperatures. Figures 5(a) and 5(b) compare the linear shrinkage strain of each powder type in the X and Z directions. In both cases, shrinkage increases with temperature, reflecting progressive densification. However, MIM-cut powders show a more pronounced directional dependence: the Z-direction strain (ε_z) consistently exceeds the X-direction (ε_x), with the gap widening at higher temperatures. At 1370 °C, ε_z reaches approximately 17.8 %, while ε_x remains around 12.6%. In contrast, LPBF-cut powders exhibit a smaller difference between directions, with ε_z and ε_x reaching approximately 15% and 12.4%, respectively, at 1390 °C. Figure 5(c) quantitatively expresses this anisotropy by presenting the Anisotropic Shrinkage Ratio (ASR = $\varepsilon_z/\varepsilon_x$) as a function of temperature. For MIM-cut powders, ASR increases steadily with temperature, reaching values above 1.4, indicating significant anisotropic shrinkage. Conversely, LPBF-cut powders show a much smaller increase in ASR, remaining close to 1.2 even at elevated temperatures, suggesting a more isotropic shrinkage behavior. Polynomial fits describe these trends well, with high correlation coefficients ($R^2 \approx 0.97\text{--}0.98$), emphasizing a strong temperature dependence of directional shrinkage.

These results demonstrate that MIM-cut powders are more prone to anisotropic sintering shrinkage, likely due to their narrower particle size distribution and lower initial green density, which promotes a higher amount of shrinkage (from 56% to 97 %). In contrast, the broader PSD and highly compact green structure of LPBF-cut powders produce lower shrinkage (58 % to 96 %). This indicates that anisotropic sintering in BJ is primarily controlled by the amount of shrinkage,

and thus by the initial and final densities of the parts. In this case, a difference of only 3 % part shrinkage between MIM and LPBF –cut powder increases sintering anisotropy by 17%.

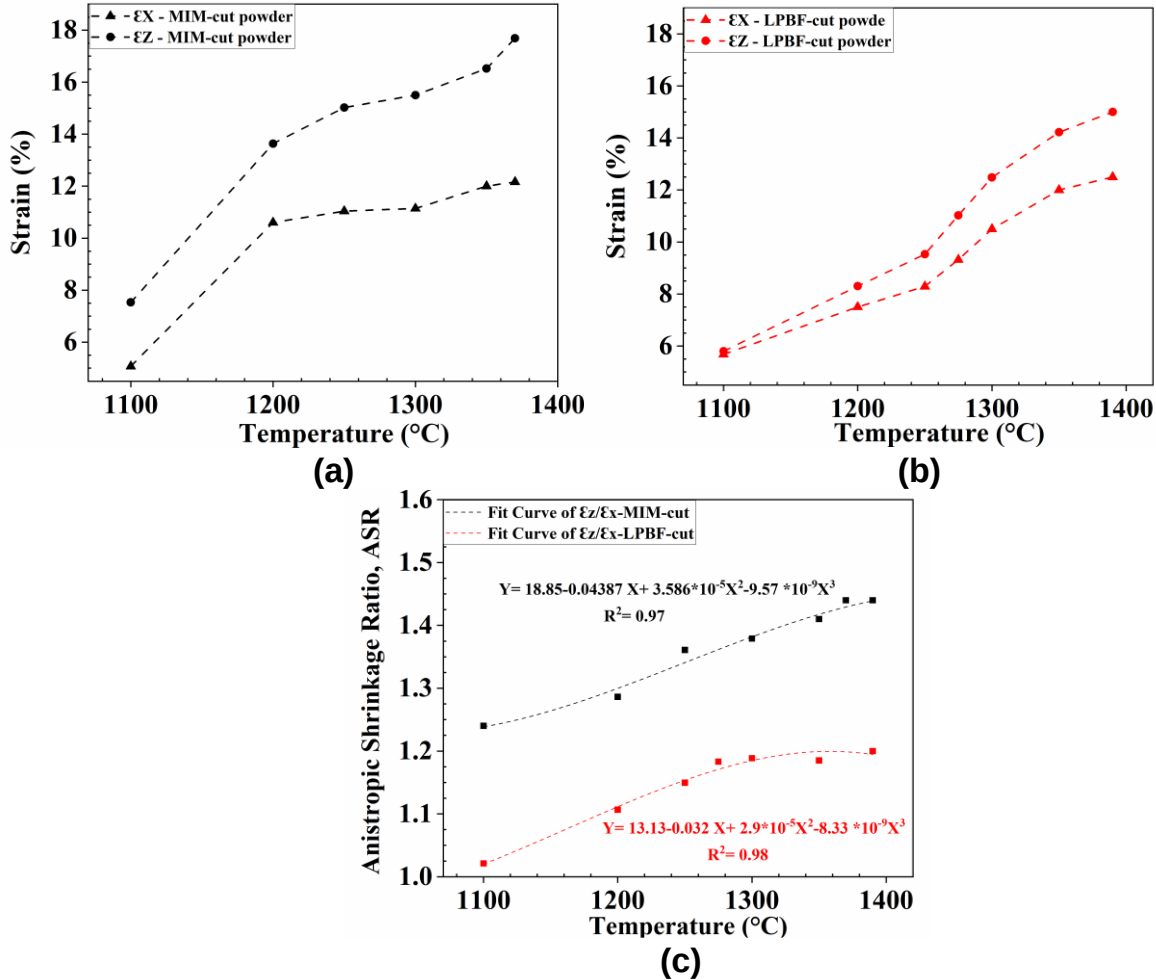


Figure 7-5 (a) Sintering strain along the X- and Z-directions for MIM-cut powder, (b) Sintering strain along the X- and Z-directions for LPBF-cut powder, (c) Anisotropic shrinkage ratio (ASR) for MIM-cut powder and LPBF-cut powder.

7.4 Conclusion

This study aimed to comprehensively evaluate the influence of powder size distribution (PSD) on the shrinkage behavior, microstructure, and densification mechanisms in BJ-316L stainless steel parts. Two powder types, MIM-cut with a narrower PSD and LPBF-cut with a broader PSD, were compared through dilatometry and SEM imaging across a range of sintering temperatures. The major conclusions are:

- 1- MIM-cut powders demonstrated greater linear shrinkage ($\sim 17.8\%$ in the Z-direction at 1370°C) and more efficient densification at lower temperatures, attributed to their finer particle size and uniform distribution. In contrast, LPBF-cut powders, with their broader PSD and higher compact packing, exhibited lower shrinkage ($\sim 15\%$ in Z) and delayed densification, requiring higher temperatures to reach comparable densities.
- 2- MIM-cut samples exhibited finer, more equiaxed grain structures ($\sim 46\text{--}48\ \mu\text{m}$) than LPBF-cut samples ($\sim 50\text{--}53\ \mu\text{m}$). This refinement is attributed to a finer PSD, which promotes faster densification, enhances grain boundary pinning, and limits thermal exposure.
- 3- The anisotropic shrinkage was more pronounced in MIM-cut samples, with an ASR exceeding 1.4, likely due to directional particle alignment during printing. LPBF-cut samples, benefiting from a broader PSD and more isotropic packing, exhibited nearly uniform shrinkage behavior ($\text{ASR} \sim 1.2$), which supports better dimensional control in sintering.

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CHAPTER 8 ARTICLE 4: Dynamic Compression Behavior of Binder-Jetted 316L Stainless Steel under Crash-Relevant Conditions

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Abstract

Understanding the high-strain-rate mechanical response of structural materials is essential for ensuring the crashworthiness of automotive components. High-strain-rate compression testing provides an effective approach for simulating crash-relevant conditions and characterizing a material's dynamic response. This study investigates the compressive behavior of binder-jetted (BJ) 316L stainless steel under high-strain-rate loading. A combined experimental–simulation approach was employed to examine the effects of temperature, strain rate, powder size distribution (PSD), and hot isostatic pressing (HIP) on the dynamic compression behavior of BJ components. BJ-316L parts were experimentally characterized using a Split Hopkinson Pressure Bar (SHPB) setup with varying strain rates (500 to 2000 s⁻¹) and temperatures (RT-600 °C) conditions. BJ specimens exhibited strain-rate strengthening and pronounced thermal softening under dynamic compression. At room temperature, peak flow stress increased by 57% (380 MPa to 900 MPa) as the strain rate increased from 500 to 2000 s⁻¹. At a strain rate of 2000 s⁻¹, increasing the temperature to 600 °C reduced peak stress by 60 % (900 MPa to 364 MPa), reflecting thermally activated dislocation motion and enhanced dynamic recovery. Compared with wrought 316L, the binder-jetted specimens exhibited 10% lower peak stresses, while both materials showed similar strain-rate hardening and flow-curve characteristics, suggesting comparable rate-controlling deformation mechanisms. PSD and HIP have been found to have minimal influence on dynamic compression behavior. Strain rate and temperature are found to impact microstructure significantly. Increasing strain rate promotes dislocation accumulation, leading to higher misorientation, dislocation density, and subgrain formation, whereas elevated temperatures activate dynamic recovery, which reduces misorientation and weakens crystallographic texture. The constitutive models, including Johnson–

Cook, modified Johnson–Cook, Hensel–Spittel, and modified Hensel–Spittel, were calibrated against experimental data. The modified Hensel–Spittel model demonstrated the best predictive capability ($R^2 = 0.951$), yielding the lowest average absolute relative error (AARE) of 5.49% across the entire temperature–strain-rate domain. Overall, the results comprehensively characterize the dynamic compression behavior of BJ-316L parts, establishing their suitability for fabricating automotive parts with high crashworthiness.

Keywords: Binder jet additive manufacturing; 316L stainless steel; High strain-rate deformation; Powder size distribution; Hot isostatic pressing; Constitutive modeling.

8.1 Introduction

Binder jetting (BJ) is an additive manufacturing (AM) process used to fabricate complex geometry 3D metal structures [1]. Parts are printed by depositing a liquid binding agent layer by layer using computer-aided design (CAD) [2]. Subsequently, the printed parts are subjected to a sintering heat treatment to produce fully dense, near-net-shape components [3,4]. Compared to other AM processes, BJ is cost-effective [224] and offers a higher production rate (100X quicker) [4], making it appealing for high-throughput applications such as medical, aerospace, defense, and automotive [3,4,25,34].

One of the key challenges in the automotive industry is high–strain-rate loading during crash and impact events [225,226], where materials are subjected to high strain rates (10^2 – 10^3 s⁻¹) and localized temperature rise due to adiabatic heating [227–230]. Under such conditions, deformation mechanisms differ significantly from those under quasi-static loading [229,231]. Strain-rate hardening, thermal softening, and dynamic recovery govern the evolution of flow stress and deformation stability [229,232,233]. Therefore, a comprehensive understanding of high-strain-rate behavior is essential for assessing the suitability of additively manufactured components in crash-relevant applications.

Austenitic stainless steel 316L is widely used in structural and automotive applications due to its excellent combination of high corrosion resistance and mechanical properties [101,102]. 316L is well-suited for the BJ process due to its good printability, low susceptibility to cracking, and a stable γ -austenitic microstructure [55,224]. Metallurgically, its relatively medium stacking-fault energy (20–40 mJ/m²) promotes deformation dominated by dislocation slip and subgrain formation

rather than martensitic transformation, which is advantageous for stable energy absorption under dynamic loading [229,234].

Previous researchers have focused on investigating the dynamic mechanical behavior of stainless steels produced by conventional and laser-based additive manufacturing routes [229,235]. W. Lee et al. [236] reported that wrought 316L stainless steel exhibits pronounced strain-rate sensitivity, with the flow stress increasing significantly with increasing strain rate during high-strain-rate deformation. Similarly, Lei et al. [237] reported pronounced strain-rate sensitivity and significant thermal softening, attributed to dislocation multiplication, interaction, and dynamic recovery in LPBF-316L. Some works have explored the influence of build direction, heat treatment, and temperature on the dynamic response of laser powder bed fusion (LPBF) 316L [227,238]. However, no focused research has investigated the dynamic mechanical response of 316L parts fabricated using the BJ process.

Dynamic compression is a multi-factor phenomenon that requires careful optimization of process parameters, including strain rate, temperature, and material microstructure [226]. High-strain-rate compression testing using the Split Hopkinson Pressure Bar (SHPB) technique provides a reliable method for characterizing material behavior under crash-relevant conditions [239,240]. SHPB enables controlled testing at strain rates comparable to real automotive impact events while allowing simultaneous evaluation of temperature effects [227,241].

Constitutive modeling helps accelerate the optimization of test parameters by eliminating cost- and time-intensive experimentation. Phenomenological models such as Johnson-Cook [242–245] and Hensel-Spittel [237,246,247] have been widely used to describe high-strain-rate deformation of metals, establishing the coupled effect of plastic strain, strain rate, and temperature. While these models have been calibrated for wrought and LPBF materials [231,248], their predictive capability for binder-jetted 316L under combined high-temperature and high-strain-rate conditions remains unexplored.

Several critical knowledge gaps persist regarding the dynamic behavior of binder-jetted 316L stainless steel. High-strain-rate compressive response of BJ-316L across a wide temperature-strain rate domain relevant to crash conditions remains insufficiently understood. Moreover, the effects of PSD and the post-processing technique (HIP) on dynamic compression need to be addressed.

The present study aims to address the identified gaps by investigating the high-strain-rate compressive behavior of BJ-316L by examining the influence of strain rate (500-2000 s⁻¹), temperature (25-600 °C), powder size distribution (MIM-cut and LPBF-cut), and hot isostatic pressing (HIP). The effect of dynamic compression on microstructure has been established through a detailed analysis of phase evolution, grain structure and orientation, grain boundaries, misorientations, and dislocations. Finally, the four constitutive models (Johnson-Cook, modified Johnson-Cook, Hensel-Spittel, and modified Hensel-Spittel) have been calibrated for BJ-316L. The proposed integrated experimental-modeling framework advances current understanding of thermo-viscoplastic deformation in BJ-316L and provides predictive tools to assess its suitability for crash-relevant automotive applications.

8.2 Experimental procedures

8.2.1 Material

Gas-atomized austenitic stainless steel 316L powders with two different particle size distributions (PSD) were used in this study: (i) metal injection molding (MIM-cut) and (ii) laser powder bed fusion (LPBF-cut). Fig. 1 presents the powder morphology obtained by scanning electron microscopy (SEM) and the particle size distribution for both powders, measured with a CAMSIZER X2 system (RETSCH, Wuppertal, Germany). Fig. 1a shows that the MIM-cut powder exhibits a relatively narrow PSD, with D10 = 6.1 μm, D50 = 12.2 μm, and D90 = 19.8 μm. In contrast, the LPBF-cut powder shows a broader size distribution (Fig. 1b), with D10 = 9.1 μm, D50 = 20.3 μm, and D90 = 40.1 μm, indicating a wider particle-size range. Morphology in Fig. 1 shows that both powders consist predominantly of spherical gas-atomized particles, with the LPBF-cut powder containing larger particles and a wider size distribution. The nominal chemical composition of the 316L powder is provided in Table 1.

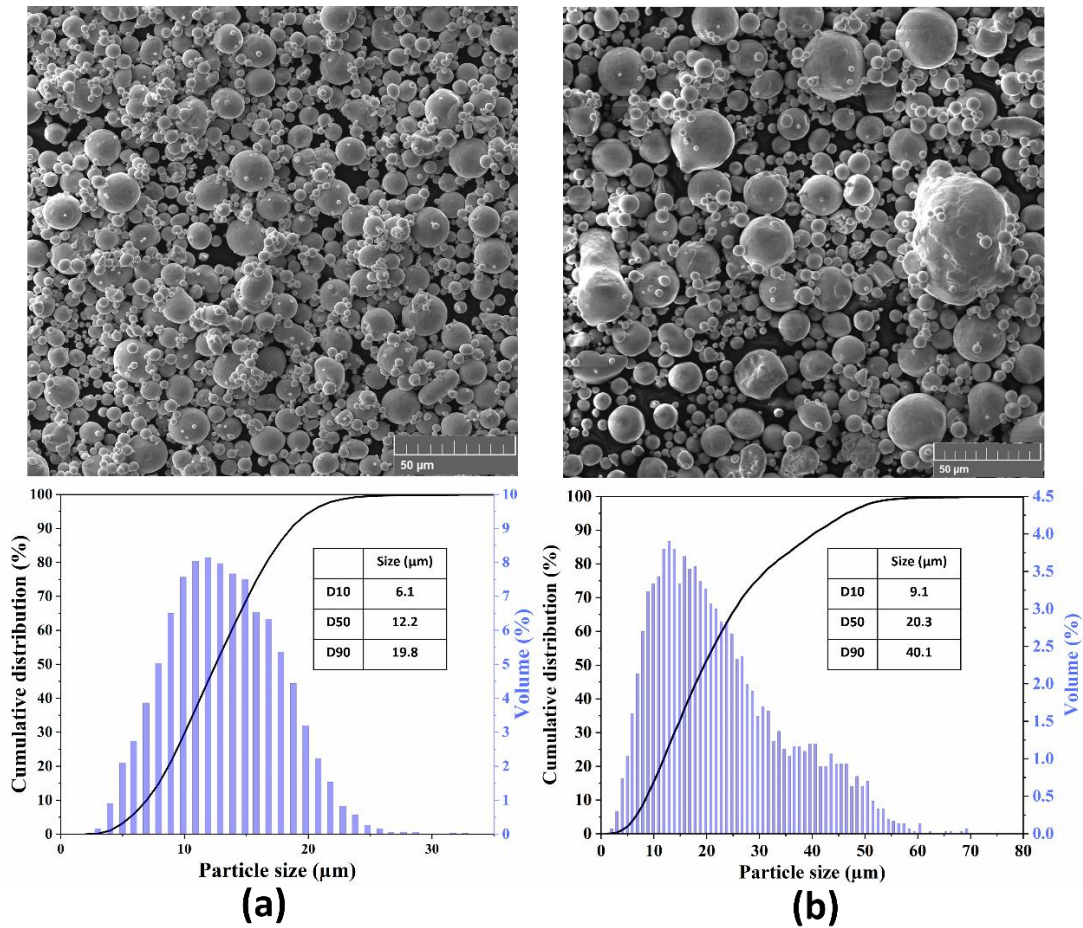


Figure 8-1 Morphology and PSD of 316L (a) MIM-cut powder and (b) LPBF-cut powder.

Table 8.1 Chemical composition of gas atomized SS 316L powder (wt. %).

Cr	Ni	Mo	Mn	Si	C	O	S	Fe
16-18	10-14	2-3	≤ 2.0	≤ 1.0	≤ 0.03	≤ 0.14	≤ 0.04	Bal.

8.2.2 Binder jetting process

Cuboidal specimens were printed using a commercial Exone Innovent+ binder jet printer and water-based binder (Exone AquaFuse). The binder is primarily a glycol-based solvent combined with proprietary additives designed to enhance particle adhesion and improve flow characteristics [178]. Two powder feedstocks produced via LPBF-cut and MIM-cut routes were employed to evaluate the effect of particle size distribution (PSD) on dynamic mechanical behavior. The binder jet printing parameters used for part printing are summarized in Table 2. The LPBF-cut samples

exhibited green densities of approximately 58–60%, whereas slightly lower green densities of 56–57% were obtained for the MIM-cut samples.

Post printing, the green parts were thermally cured at 180 °C for 6 h to remove residual binder solvent. Thermal debinding was subsequently performed in a hydrogen (H₂) atmosphere at 600 °C for 6 h to remove the organic binder. Sintering was performed in a pure- H₂ reducing atmosphere using a horizontal tube furnace. The sintering cycle consisted of heating at 5 °C/min to a peak temperature of 1370 °C for the MIM-cut samples and 1390 °C for the LPBF-cut samples, followed by an isothermal hold for 6 h. After sintering, the specimens were cooled to room temperature at 5 °C/min under the same reducing atmosphere. The sintered density was measured using the Archimedes method in accordance with ASTM B962-13 [219]. Selected samples were further subjected to hot isostatic pressing (HIP) at 1163 ± 14 °C under 15,000 ± 500 psi for 4 h ± 15 min.

Table 8.2 Binder jet printing parameters for stainless steel 316L.

Parameter	Value
Binder saturation (%)	100
Binder set time (s)	10
Bed temperature (°C)	65
Recoating speed (mm/s)	90
Layer thickness (μm)	100

8.2.3 Dynamic compression testing

For dynamic compression experiments, the specimens were machined into cylindrical geometries with a height and diameter of 4 mm each. The loading axis of each specimen was oriented perpendicular to the build (printing) direction. High-strain-rate compression tests were conducted using a Split Hopkinson Pressure Bar (SHPB) system equipped with an infrared heating furnace. Dynamic compression was performed at five different temperatures: 25 °C, 150 °C, 300 °C, 450 °C, and 600 °C. Post-deformation, the specimens were ramped to the target temperature at 3 °C/s with a 5 min dwell time to ensure thermal equilibrium. Thermocouples were adequately placed to monitor and report temperature with high accuracy. To minimize any associated uncertainty and

ensure repeatability in temperature measurement, multiple calibrations were performed. During testing, a quartz tube was used to thermally insulate the specimen from the surrounding environment, thereby reducing heat loss. The imposed strain rates were regulated by adjusting the pressure applied to the pneumatic launch cylinder of the SHPB system (Fig. 2), with operating pressures ranging from 10 to 100 psi. Dynamic compression experiments were conducted at nominal strain rates of 500, 1000, and 2000 s^{-1} . The achieved strain rate depends on the launch cylinder pressure and the testing temperature; hence, a series of calibration experiments was performed to determine appropriate pressure levels for each testing condition. The resulting cylinder pressures required to attain the target strain rates at different temperatures are summarized in Table 3. During testing, strain signals were measured using half-Wheatstone bridge configurations mounted on the incident and transmitted bars and recorded through a high-speed data acquisition system. The overall SHPB experimental configuration is schematically illustrated in Fig. 2.

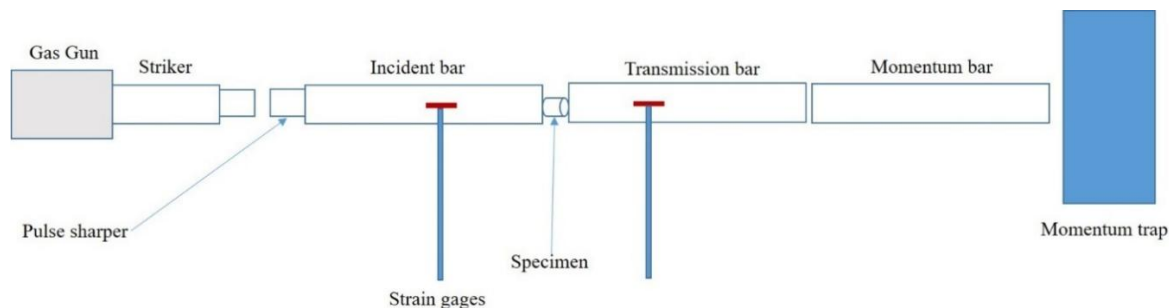


Figure 8-2 Schematic illustration of the SHPB setup used for high-strain-rate compression testing. The voltage–time signals recorded during SHPB testing were used to calculate the specimen's stress–strain response. The measured incident, reflected, and transmitted voltages were converted into strain, strain rate, and stress using standard SHPB relations as outlined in Eqs. (1)– (4) [99,249], together with the specimen dimensions and the known mechanical properties of the pressure bars. Engineering stress and strain were first obtained assuming a constant cross-sectional area. They were subsequently converted to true stress and true strain to account for geometric changes during deformation. These true stress–strain data were used to evaluate the material's dynamic mechanical response at elevated temperatures.

Table 8.3 Cylinder pressure levels during the Split Hopkinson Pressure Bar (SHPB) test at different test temperatures and imposed strain rates.

Temperature (°C)	Cylinder pressure at different strain rates (psi)		
	500 s ⁻¹	1000 s ⁻¹	2000 s ⁻¹
Room Temperature (RT)	12	22	62
150	12	22	48
300	12	20	42
450	10	18	40
600	10	18	40

$$\varepsilon_n(t) = \frac{2v_n(t)}{2v_{input}\phi} \quad (1)$$

$$\dot{\varepsilon}_s = \frac{\sqrt{(E_b/\rho_b)}^{-1}}{L_s} \varepsilon_r \quad (2)$$

$$\varepsilon_s = 2\dot{\varepsilon}_s \frac{L_s}{\sqrt{E_b/\rho_b}} \varepsilon_t \quad (3)$$

$$\sigma_s = E_b \frac{A_b}{A_s} \varepsilon_r \quad (4)$$

8.2.4 Material constitutive modelling

The dynamic stress–strain responses measured using the SHPB apparatus, as described in Section 2.3, were employed to develop four material constitutive models: the Johnson–Cook [250], modified Johnson–Cook [245,251,252], the Hensel–Spittel [253], and the modified Hensel–Spittel [254,255]. For constitutive calibration, the yield stress was defined as the stress at a strain of 0.01, consistent with values obtained using the conventional 0.2% offset method [99].

The Johnson–Cook (JC) constitutive model describes the flow stress as a multiplicative function of strain hardening, strain-rate sensitivity, and thermal softening, assuming these three effects are independent. The JC flow stress (σ) is expressed by Eq. (5) [250], while the homologous temperature (T^*) is defined by Eq. (6).

$$\sigma = (A + B\varepsilon^n)[1 + C \ln(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0})] (1 - T^{*m}), \quad (5)$$

$$T^* = \frac{T - T_{ref}}{T_m - T_{ref}} \quad (6)$$

Where σ is the equivalent flow stress, ε is the equivalent plastic strain, $\dot{\varepsilon}$ is the strain rate, and parameter A is the yield stress at the reference strain rate ($\dot{\varepsilon}_0$) and reference temperature (T_{ref}). B and n define the magnitude and exponent of strain hardening, and C governs the strain-rate sensitivity. The homologous temperature (T^*) accounts for thermal softening and is defined using the current temperature T, the reference temperature (T_{ref}), and the melting temperature (T_m). The JC formulation captures the overall influence of strain rate and temperature on the flow behavior; however, the lack of coupling between plastic strain and temperature limits its ability to reproduce the nonlinear stress–strain curvature observed at elevated temperatures and large strains in binder-jetted 316L.

The modified Johnson–Cook (m-JC) model improves upon this limitation by introducing temperature-dependent strain-hardening parameters, thereby enabling partial coupling between deformation and thermal effects. To address this, two key modifications are introduced; first, the original power-law strain-hardening term ($A + B\varepsilon^n$) is replaced by a quadratic strain-dependent function ($A_1 + B_1\sigma\varepsilon + B_2\varepsilon^n$), providing greater flexibility to reproduce the experimental stress–strain response within the industrially relevant strain range, second, the classical homologous

temperature softening term $(1 - T^{*m})$ is replaced by an exponential temperature term coupled with strain rate $([\lambda_1 + \lambda_2 \ln(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0})] (T - T_{ref}))$, introducing explicit thermomechanical coupling. This new term allows thermal softening to evolve with both temperature and strain rate, capturing the combined effects of adiabatic heating and dynamic deformation that are not represented in the original JC model. Accordingly, the flow stress in the modified Johnson–Cook (m-JC) model is expressed as [245,251,252]:

$$\sigma = (A_1 + B_1 \sigma \varepsilon + B_2 \varepsilon^n) [1 + C \ln(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0})] ([\lambda_1 + \lambda_2 \ln(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0})] (T - T_{ref})), \quad (7)$$

The parameters A_1 , B_1 , and B_2 govern the modified strain-hardening behavior, C controls strain-rate sensitivity, and λ_1 and λ_2 describe the coupled influence of temperature and strain rate relative to the reference temperature T_{ref} .

The Hensel-Spittel (HS) model, in which strain, strain rate, and temperature are intrinsically coupled. The HS flow stress is expressed as [253]:

$$\sigma = A e^{m_1 T} e^{m_2 \dot{\varepsilon}^{m_3}} e^{m_4 / \varepsilon} (1 + \varepsilon)^{m_5 T} e^{m_7 \varepsilon \dot{\varepsilon}^{m_8} T^{m_9}} \quad (8)$$

Where σ is the equivalent flow stress, ε is the equivalent plastic strain, and $\dot{\varepsilon}$ is the plastic strain rate. The parameters A , m_1 - m_8 are material constants, and T is absolute temperature. The HS formulation provides a more physically consistent description of high-temperature and high-strain-rate deformation and results in improved prediction accuracy compared to JC-based models across the investigated conditions[99].

The modified Hensel–Spittel (m-HS) model further enhances this framework by introducing additional thermo-viscoplastic coupling, which refines the representation of strain-hardening curvature and temperature–strain interactions. In the modified formulation (Eq. 9), the flow stress is expressed as a hyperbolic sine function [254].

$$\sinh(\alpha \sigma) = A e^{m_1 T} e^{m_2 \dot{\varepsilon}^{m_3}} e^{m_4 / \varepsilon} (1 + \varepsilon)^{m_5 T} e^{m_7 \varepsilon} \quad (9)$$

where α is an additional material parameter introduced in the modified model to capture nonlinear stress dependence at elevated strain rates. The hyperbolic sine term provides a smoother, more

accurate representation of flow behavior over a wide stress range, particularly at high strain rates and elevated temperatures, where the classical HS model tends to over- or under-predict stress.

1. 2.5. Microstructure characterization

Microstructure characterization samples were prepared using standard metallographic procedures, including grinding with SiC papers (320, 600, 800, and 1200 grit), followed by diamond polishing with 6 μ m, 3 μ m, and 1 μ m suspensions, and then final polishing with 0.04 μ m colloidal silica. Polished samples were then cleaned ultrasonically in distilled water to remove residual debris. Optical microscopy was performed using a Keyence VHX-7000 digital microscope to evaluate porosity and measure grain size in green and sintered samples. The average grain size was determined using the ASTM E112–13 line-intercept method based on at least three micrographs per condition. Microstructural features, including pore morphology, grain boundaries, and phase constituents, were further examined using a JEOL JSM-7600F field-emission scanning electron microscope (FEG-SEM) equipped with an Oxford Instruments X-Max 80 mm² EDS detector. Electron backscatter diffraction (EBSD) analysis was conducted using a JEOL JSM-IT800 scanning electron microscope with an Oxford Instruments EBSD system, operating at 20 kV and 25 nA with a step size of 75 nm. EBSD data were processed using AZtec-Crystal software to generate phase maps, inverse pole figures (IPFs), pole figures (PFs), grain-boundary statistics, dislocation density, and kernel-average misorientation (KAM) maps.

8.3 Results and discussion

8.3.1 Initial microstructure

Fig. 3 shows low- and high-magnification SEM micrographs of BJ 316L parts fabricated using LPBF-cut and MIM-cut powders in the as-sintered and HIP-treated conditions. In all cases, the microstructure is predominantly composed of equiaxed γ -austenite (FCC) grains with annealing twin boundaries within the austenitic grains, a low percentage of δ -ferrite (BCC), and limited residual porosity. The as-sintered LPBF-cut specimens (Fig. 3a–b) exhibit an average grain size of $45 \pm 3 \mu\text{m}$ and a final density of approximately 98.3%. Minor δ -ferrite regions appear as darker contrast areas, primarily along grain boundaries and triple junctions. In comparison, the as-sintered MIM-cut specimens (Fig. 3e–f) show a finer and more homogeneous grain structure with an average grain size of $37 \pm 2 \mu\text{m}$ and a slightly higher final density of $\sim 98.8 \%$. The refined microstructure in MIM-cut powder is attributed to the higher specific surface area and smaller

interparticle pore size associated with narrow PSD powders, which enhance diffusion-controlled mass transport and accelerate early-stage neck formation during sintering [256–258]. Consequently, pore elimination occurs more uniformly, limiting abnormal grain growth.

Following HIP treatment (Fig. 3c–d and g–h), porosity is nearly eliminated, with relative densities increasing to $\sim 99.8\%$ (LPBF-cut) and $\sim 99.9\%$ (MIM-cut). Grain coarsening becomes evident in both powder conditions. The LPBF-cut specimens reach an average grain size of approximately $57 \pm 10\ \mu\text{m}$, whereas the MIM-cut samples exhibit more pronounced grain growth, reaching $\sim 65 \pm 15\ \mu\text{m}$. Grain boundaries appear smoother and more continuous after HIP, consistent with enhanced grain-boundary migration and grain coarsening under high-temperature, high-pressure conditions. Annealing twin boundary remains visible within the austenite large grains, which is the characteristic of thermally treated FCC stainless steels and consistent with grain growth during HIP [53,214,215,259]. The greater grain coarsening in the HIPed MIM-cut specimens correlates with their lower retained δ -ferrite fraction (5%), which reduces grain-boundary pinning. In contrast, the higher δ -ferrite content (10.6 %) in LPBF-cut samples partially restrains boundary mobility, resulting in comparatively moderate grain growth [260].

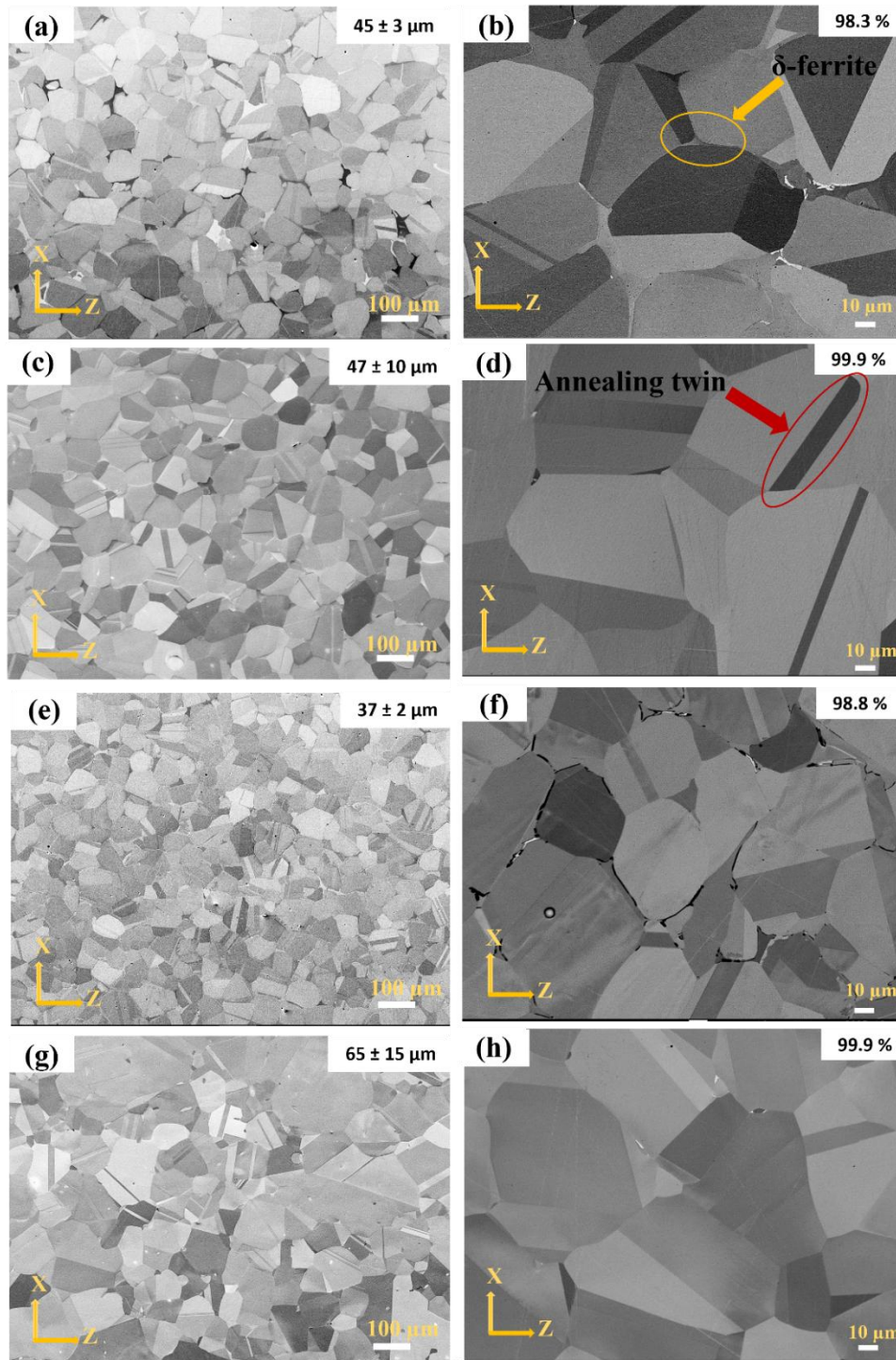


Figure 8-3 Low magnification(left)- and High-magnification (right) SEM micrographs of BJ SS 316L parts for two powder types and two processing conditions: (a–b) As-sintered LPBF-cut samples, (c–d) HIPed LPBF-cut samples, (e–f) as-sintered MIM-cut samples, and (g–h) HIPed MIM-cut samples. The average grain size (μm) and relative density (%) for each condition are indicated in the corresponding images.

Fig. 4 shows the inverse pole figure (IPF) and phase maps for all conditions. The as-sintered LPBF-cut specimen (Fig. 4a) exhibits equiaxed grains with weak crystallographic texture, reflecting extensive recrystallization during sintering [182,261]. After HIP (Fig. 4c), the grain structure becomes coarser, but the overall texture intensity remains weak, indicating that HIP promotes grain growth primarily through grain boundary migration under the high-temperature and high-pressure conditions rather than orientation selection [262]. Similar behavior is observed for MIM-cut as-sintered specimens, showing comparatively more uniform equiaxed grain microstructure with highly random grain orientation in Fig. 4e. Post HIPing, the MIM-cut printed specimens show grain coarsening with reduced randomness in the grain orientation as presented in Fig. 4g.

The phase maps for as-sintered LPBF cut specimens in Fig. 4b reveal the presence of a minor delta ferrite phase (11.54%) preferentially located along grain boundaries and in the triple junctions. However, post HIPing, a slight reduction in delta ferrite (10.58%) is observed in Fig. 4d. A similar trend of reduction in δ -ferrite from 7.98% in as-sintered to 5.08% post HIPing has been observed for the MIM-cut specimens, as shown in Fig. 4(f, h). Exposure to high temperature during HIPing leads to δ -ferrite transforming to the σ phase, as evidenced by increases in the σ -phase proportion from 0.04% to 0.15% and from 0.02% to 0.1% for the LPBF-cut and MIM-cut powders, respectively. High-temperature exposure in HIP also leads to redistribution of γ -stabilizing elements, which destabilize the retained δ -ferrite, promoting its dissolution and a strong diffusion-driven δ - γ transformation [262].

Fig.5 presents the $\{100\}$, $\{110\}$, and $\{111\}$ pole figures of γ -phase for all samples. The pole figure of the LPBF-cut, as-sintered sample in Fig. 5a exhibits weak texture with maxima of low intensity distributed in $\{100\}$, $\{110\}$, and $\{111\}$, indicating significantly random orientation distribution. Post-HIP treatment, the LPBF-cut sample maintains a weak texture, and slight sharpening of maxima is observed in $\{100\}$, indicative of limited orientation selection during grain growth [262,263]. No strong $\{100\}$, $\{110\}$, or $\{111\}$ fiber components are visible in Fig. 4b, suggesting that HIP promotes grain boundary migration and phase homogenization with minimal impact on texture strengthening [263].

The pole figures of MIM-cut specimens (Fig.5c) indicate a weak overall texture and a relatively uniform intensity distribution, compared to LPBF-cut samples. The LPBF-cut powder exhibits higher green packing density (58–60%), resulting in lower total shrinkage and reduced anisotropic

strain during sintering. This more compact initial structure promotes slightly stronger preferred orientation during grain growth, leading to marginally higher texture intensity [264,265]. In contrast, the MIM-cut condition with a lower green packing density (56–58%) exhibits greater overall shrinkage and stronger anisotropic deformation, leading to more random grain rearrangement and, consequently, weaker texture intensity.

For the MIM-cut specimens, the pole-figure intensity increases after HIP compared with the as-sintered condition, as shown in Fig.5 (c-d). The maximum intensity values in the HIPed MIM-cut condition are higher than those of the corresponding LPBF-cut sample; however, the intensity remains broadly distributed. The more pronounced increase in intensity in the HIPed MIM-cut specimens correlates with the significant grain growth observed in the IPF maps. This behavior suggests that grain growth occurred under relatively high, homogeneous grain-boundary mobility conditions. The lower retained δ -ferrite fraction in the MIM-cut condition reduces Zener pinning pressure at grain boundaries, allowing more extensive curvature-driven grain growth [260,263]. As grains coarsen, a mild orientation difference develops due to differential boundary mobility between neighboring grains, resulting in moderate intensity enhancement without strong crystallographic texture [266]. In contrast, the HIPed LPBF-cut specimens exhibit comparatively smaller grain growth and only limited texture evolution, likely due to the higher retained δ -ferrite fraction, which partially restricts grain-boundary migration. Overall, the pole figure analysis confirms that HIP promotes grain growth in both powder conditions, with more pronounced grain coarsening and moderate texture sharpening in the MIM-cut specimens, with a weak γ -phase texture.

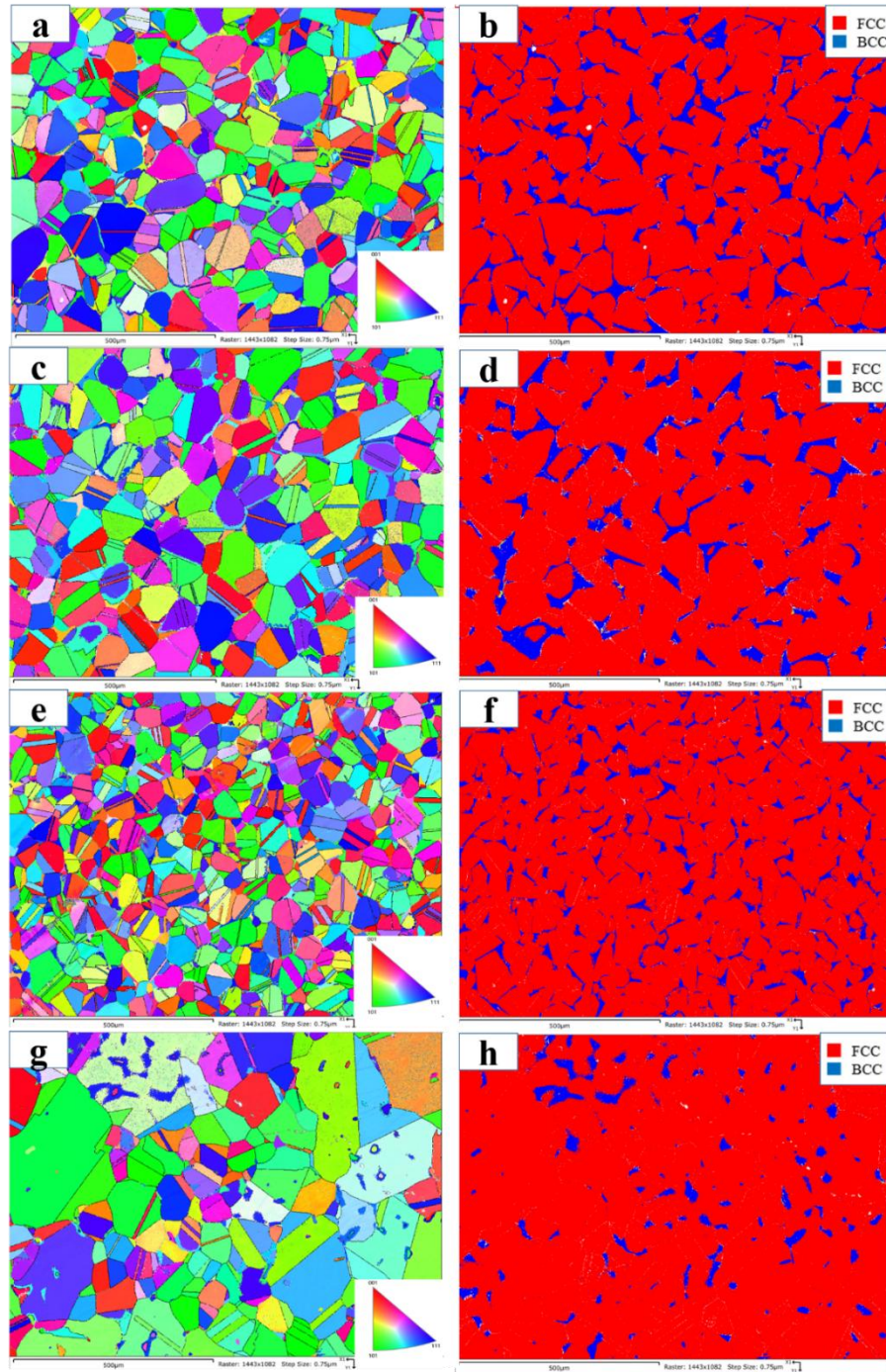


Figure 8-4 Inverse pole figure (IPF) and phase maps (FCC/BCC) of binder-jetted 316L parts printed using different PSDs and post-processing treatments: (a, b) LPBF-cut, as-sintered; (c, d) LPBF-cut, HIPed; (e, f) MIM-cut, as-sintered; (g, h) MIM-cut, HIPed.

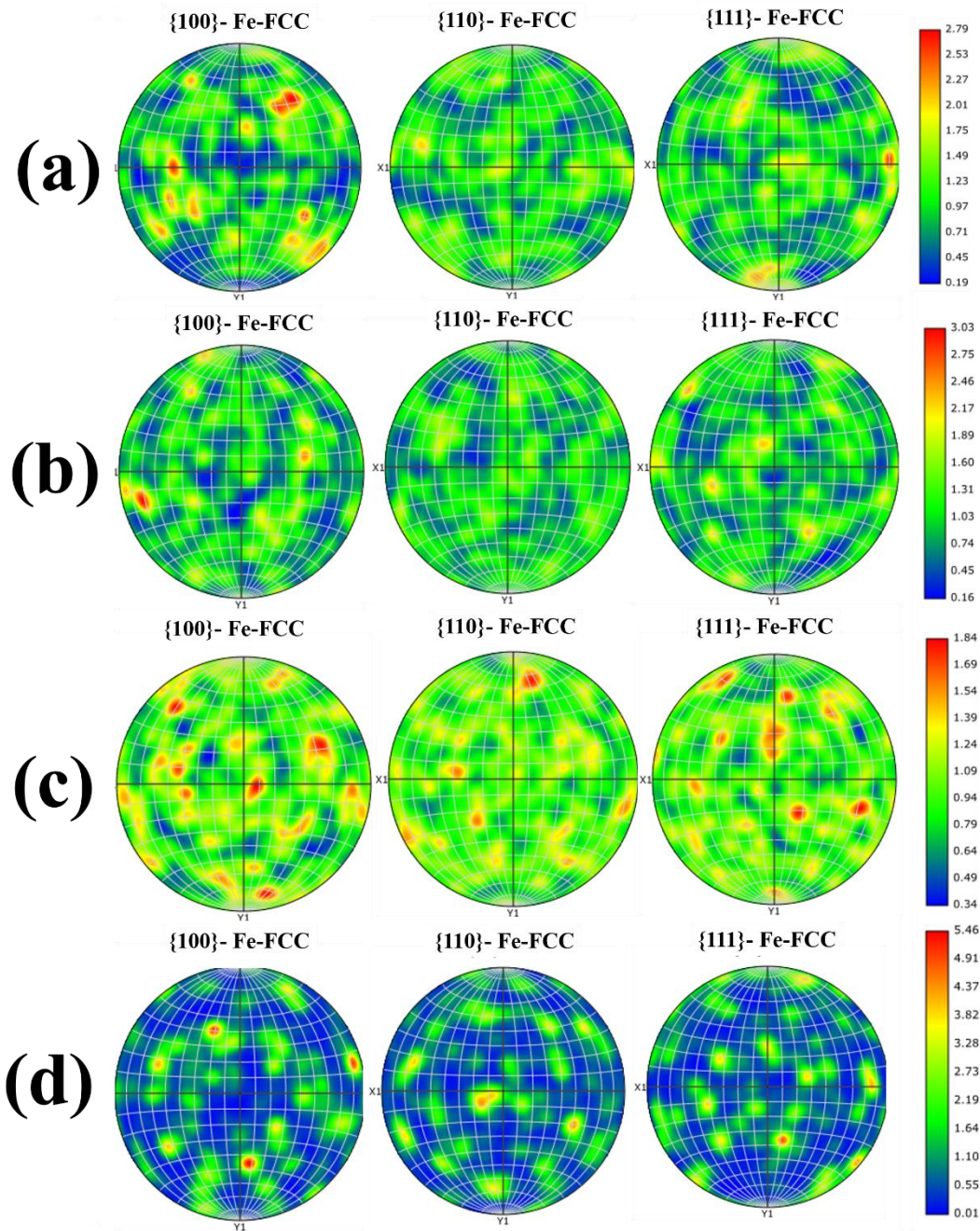


Figure 8-5 Pole figure of binder-jetted 316L parts : (a) LPBF-cut, sintered; (b) LPBF-cut, HIPed; (c) MIM-cut, sintered; (d) MIM-cut, HIPed.

8.3.2 True stress–strain curves and dynamic response curves

Fig. 6 presents the true stress–true strain response of (a) binder-jetted (as-sintered) and (b) wrought 316L part compressed using the Split Hopkinson Pressure Bar (SHPB) at strain rates of 500, 1000, and 2000 s^{-1} and at temperatures of room temperature (RT), 150, 300, 450, and 600 $^{\circ}\text{C}$. Each curve

represents the evolution of true stress as a function of true strain at a specific temperature. The curve shows that for both materials, flow stress decreases with increasing temperature, indicating pronounced thermal softening. The wrought alloy exhibits higher flow stresses and stronger strain hardening across all strain rates and temperatures. In contrast, the binder-jetted material demonstrates reduced peak stress and lower hardening capacity, which can be attributed to residual porosity and microstructural variations introduced during sintering. The difference in dynamic strength becomes particularly evident at room temperature and higher strain rates, highlighting the strong effect of the processing route on mechanical response under dynamic loading.

For the BJ parts (Fig. 6a), at all strain rates, the peak stress decreases by approximately 50–60% as the temperature increases from RT to 600 °C. Increasing the strain rate from 500 to 2000 s⁻¹ raises the peak stress by ~57% at RT (380 MPa- 900 MPa) and ~48% (187 MPa- 364 MPa) at 600 °C, indicating moderate strain-rate sensitivity. The curves exhibit continuous work hardening up to ~0.15–0.20 true strain followed by gradual saturation, with temperature exerting a stronger effect on peak stress than strain rate. The wrought 316L alloy exhibits similar qualitative trends but consistently higher flow stresses across all temperatures and strain rates. At 500 s⁻¹ and room temperature, the wrought alloy reaches 480 MPa compared to 380 MPa for the BJ material, corresponding to an increase of approximately 26%. This difference decreases with increasing strain rate, reaching ~15 % at 1000 s⁻¹ (RT) and further reducing to ~10 % at 2000 s⁻¹ (RT). Although the maximum strengths are different, both materials exhibit similar strain-hardening and saturation behavior, suggesting that the fundamental deformation mechanisms under dynamic loading are comparable.

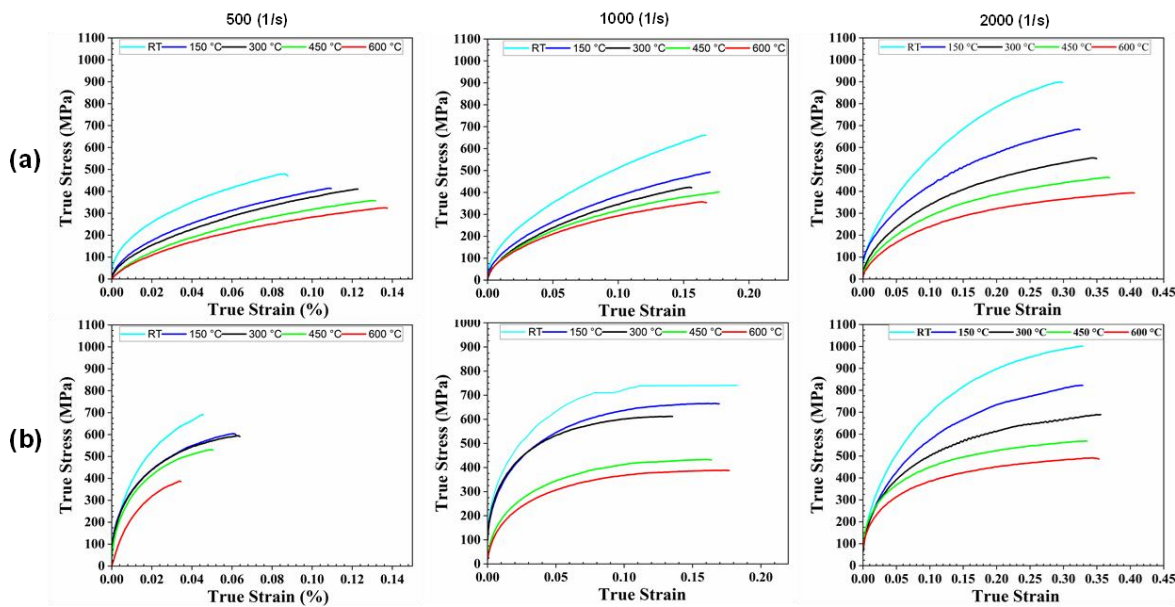


Figure 8-6 True stress–true strain curves as a function of temperature for (a) binder-jetted as-sintered and (b) wrought SS 316L subjected to SHPB tests at strain rates of 500, 1000, and 2000 s^{-1} .

The dynamic response was further analyzed in terms of yield strength, work-hardening exponent (n), and total strain across the investigated strain-rate and temperature range. The dynamic yield stress was defined as the flow stress at $\varepsilon = 0.01$, since austenitic stainless steels exhibit a gradual elastic–plastic transition without a distinct yield point under SHPB loading. This definition yields values comparable to the conventional 0.2% offset method and was used for constitutive model calibration [99]. The work-hardening coefficient describes material strengthening as deformation progresses and is calculated using the Hollomon relation ($\sigma = K\varepsilon^n$), where n quantifies the degree of strain hardening [267]. The total strain reflects the specimen's deformation capacity, whereas the maximum true stress represents the highest stress sustained during dynamic loading.

Analysis of the true stress-true strain curve for yield strength suggests that strength increases with increasing strain rates at all temperatures for both the specimens (wrought and BJ). For the binder-jetted specimen, at 25 °C, the yield strength increases from approximately 220 MPa at a strain rate of 500 s^{-1} to about 300 MPa at 2000 s^{-1} ($\approx 35\%$ increase). Comparatively, the yield strength for wrought 316L increases from nearly 250 MPa to 320 MPa over the same strain-rate range ($\approx 28\%$ increase). However, increasing the temperature significantly reduces strength, mainly due to thermal softening. At 2000 s^{-1} , raising the temperature from 25 °C to 600 °C reduces the yield strength from ~ 300 MPa to ~ 130 MPa in the BJ material ($\approx 55\text{--}60\%$ reduction), while that for

wrought decreases from ~ 320 MPa to ~ 190 MPa ($\approx 40\%$ reduction). Overall, the wrought alloy consistently exhibits higher yield strength, approximately 40–50% greater than that of the binder-jetted specimen, primarily due to its fully dense, homogeneous microstructure [268].

The work-hardening exponent (n) of BJ 316L is generally higher than that of the wrought alloy across the investigated conditions. BJ specimens exhibit n values of approximately 0.35–0.54, whereas the wrought material shows lower values of about 0.14–0.40. Increasing the strain rate tends to increase the work-hardening response. The work-hardening exponent (n) for both materials decreases with increasing temperature. For example, at a strain rate of 2000 s^{-1} , n for BJ 316L decreases from approximately 0.50 at $25\text{ }^{\circ}\text{C}$ to about 0.36 at $600\text{ }^{\circ}\text{C}$, while for the wrought alloy it decreases from approximately 0.38 to 0.20 over the same temperature range. This trend reflects the typical reduction in strain-hardening capacity at elevated temperatures, driven by enhanced dislocation recovery and thermal softening [268].

Regarding total strain, both materials exhibit increased deformation with increasing strain rate and temperature. For BJ specimens, total strain increases from ~ 0.08 – 0.13 at 500 s^{-1} to ~ 0.30 – 0.42 at 2000 s^{-1} , depending on temperature. Wrought 316L shows a similar strain-rate dependence, increasing from ~ 0.04 – 0.06 to ~ 0.32 – 0.35 over the same strain-rate range. At 2000 s^{-1} , temperature further enhances deformation, with BJ specimens increasing from ~ 0.30 at $25\text{ }^{\circ}\text{C}$ to ~ 0.42 at $600\text{ }^{\circ}\text{C}$, while wrought 316L increases moderately from ~ 0.32 to ~ 0.35 . Elevated temperatures enhance plastic deformation by reducing flow stress and increasing dislocation mobility. Conversely, higher strain rates limit time-dependent recovery processes, leading to increased dislocation accumulation and more homogeneous deformation. As a result, strain localization is delayed, allowing the material to sustain higher total strain before failure [228].

8.3.3 Effect of temperature and strain rate on dynamic response

Fig. 7 shows the variation of maximum true stress with temperature for binder-jetted (BJ) and wrought 316L stainless steel specimens subjected to dynamic compression using the Split Hopkinson Pressure Bar (SHPB) apparatus. For BJ 316L, Fig. 7a shows that the maximum true stress decreases with increasing temperature and decreasing strain rate. For example, at room temperature, the stress increases from approximately 380 MPa at 500 s^{-1} to about 900 MPa at 2000 s^{-1} , whereas at $600\text{ }^{\circ}\text{C}$ it decreases to approximately 187 MPa and 380 MPa, respectively. This decline in strength observed at elevated temperatures is attributed to thermal softening, in which

enhanced dislocation mobility and dynamic recovery reduce the resistance to plastic deformation. Conversely, the increase in strength with strain rate reflects positive strain-rate sensitivity, as rapid loading restricts recovery and promotes dislocation accumulation. The relatively low strength of the BJ material is attributed to residual porosity, incomplete particle bonding, and microstructural heterogeneity, which accelerate damage initiation and lead to premature softening. Similar behaviors have been reported for additively manufactured and powder-metallurgy stainless steels under high strain rates [101,269].

The wrought stainless steel consistently exhibits higher strength across all test conditions. At room temperature and 2000 s^{-1} , its maximum true stress reaches approximately 1000 MPa, which is nearly 100 MPa higher than that of the BJ part. Even at $600 \text{ }^{\circ}\text{C}$, where thermal softening is most severe, the wrought alloy maintains a strength of $\sim 450 \text{ MPa}$, compared with $\sim 390 \text{ MPa}$ for the BJ material. This improved response stems from its full density, stronger metallurgical bonding, and fine, homogeneous austenitic microstructure, which enable efficient load transfer, delay damage nucleation, and enhance dislocation storage under rapid loading. These factors contribute to less thermal degradation in wrought alloy. The observed strain-rate hardening is consistent with prior SHPB studies that highlight the critical roles of density and microstructural integrity in resisting high-strain-rate deformation [268–270].

The strain-rate sensitivity coefficient (S_{SR}) further quantifies the material's resistance to deformation as the applied strain rate increases. The strain rate-sensitivity coefficient (S_{SR}) is obtained using Eq. (5), where σ_1 and σ_2 are the true flow stresses measured at strain rates $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$, respectively [47]. Fig. 8 illustrates the variation of S_{SR} with temperature for both types of specimens tested between 1000 s^{-1} and 2000 s^{-1} . In both cases, S_{SR} decreases progressively with temperature, indicating that the influence of strain rate becomes less pronounced at elevated temperatures. This reduction results from enhanced dislocation climb and recovery, which facilitate plastic flow and diminish rate-dependent strengthening [237]. At lower temperatures ($25\text{--}150 \text{ }^{\circ}\text{C}$), the BJ specimen exhibits notably higher S_{SR} values ($\approx 350 \text{ MPa}$ at $25 \text{ }^{\circ}\text{C}$) than the wrought alloy ($\approx 280 \text{ MPa}$), indicating stronger rate sensitivity due to its porous, heterogeneous microstructure, which amplifies local strain accumulation under dynamic loading [271]. At intermediate and high temperatures ($\geq 300 \text{ }^{\circ}\text{C}$), both materials exhibit a marked decline in S_{SR} , and their values converge above $600 \text{ }^{\circ}\text{C}$, suggesting that deformation is governed by thermally assisted recovery and diffusion, thereby diminishing the role of strain rate [103,272]. Overall, the viscoplastic behavior

of both specimens is majorly governed by the interplay between thermal softening and strain-rate hardening. The BJ part is more strain-rate-sensitive at lower temperatures; however, the two materials exhibit similar, limited sensitivity at elevated temperatures. This confirms that at elevated temperatures, the dynamic deformation is mainly thermally controlled.

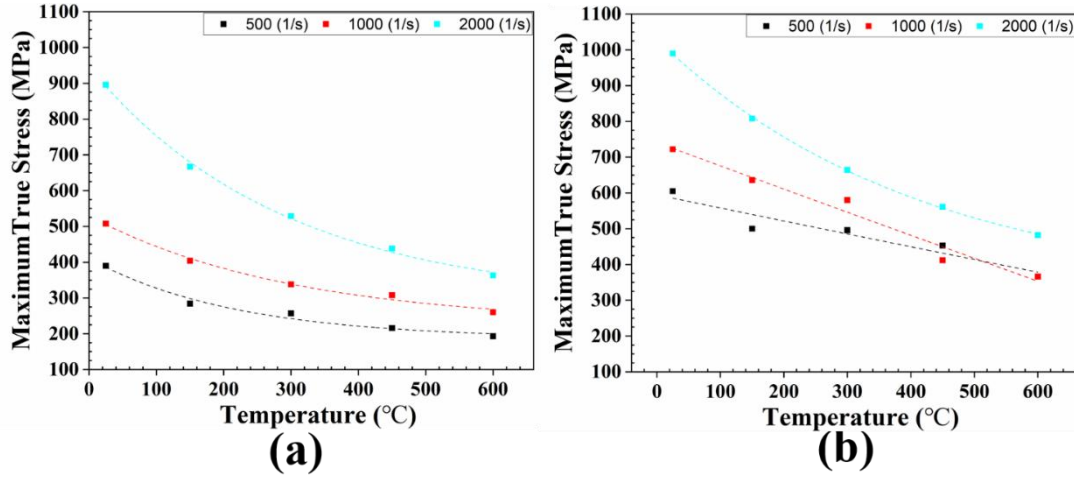


Figure 8-7 Maximum true stress versus temperature for SS 316L specimens: (a) binder-jetted and (b) wrought, obtained from SHPB tests at strain rates of 500, 1000, and 2000 s⁻¹. Maximum stresses were obtained at fixed true strains of $\epsilon = 5\%$, 10% , and 30% for strain rates of 500, 1000, and 2000 s⁻¹, respectively.

$$S_{SR} = \frac{\sigma_2 - \sigma_1}{\ln \frac{\dot{\epsilon}_2}{\dot{\epsilon}_1}} \quad (5)$$

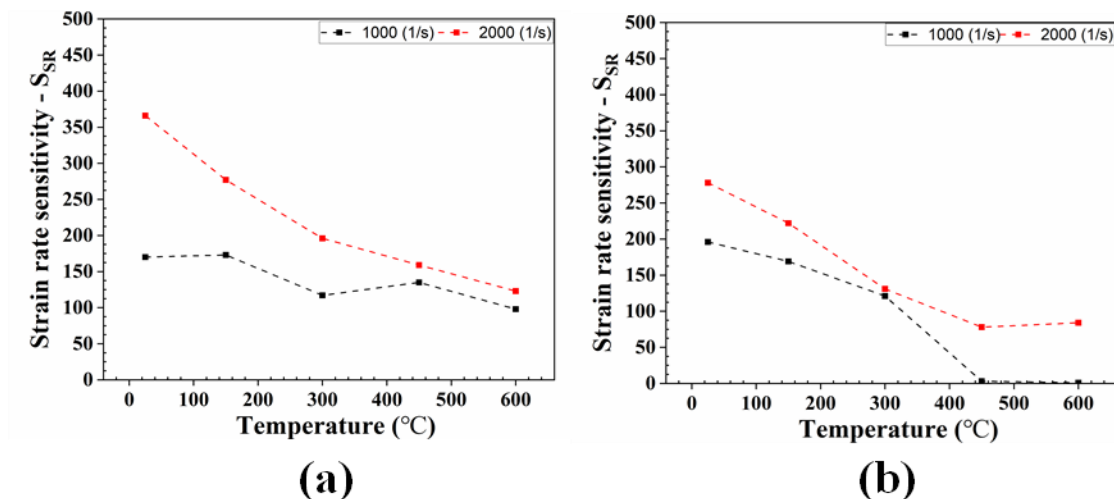


Figure 8-8 Strain-rate sensitivity as a function of temperature for SS 316L: (a) binder-jetted and (b) wrought, evaluated at strain rates of 1000 and 2000 s^{-1} .

8.3.4 Effect of temperature and high-strain-rate deformation on microstructure

Fig. 9 presents the IPF and corresponding pole figure maps (FCC), demonstrating the effects of strain rate and deformation temperature on microstructural and textural evolution. For a strain rate of 500 s^{-1} at room temperature, the IPF map in Fig. 9a reveals relatively more equiaxed grains with significant randomness in intragranular orientation. This primarily indicates plastic deformation at high strain rates, accommodated by dislocation slip in the absence of high temperatures. The corresponding pole figure maps in Fig. 9c exhibit weak maxima aligned with $\{100\}$, $\{110\}$, and $\{111\}$, indicating limited texture development even after significant plastic strain. Upon increasing the deformation temperature to 600 $^{\circ}\text{C}$ at a strain rate of 500 s^{-1} , a reduction in the orientation gradient is observed, as shown in Fig. 9b. A more homogeneous grain orientation suggests enhanced dynamic recovery at higher temperatures. Pole figures for this condition, shown in Fig. 9d, indicate weaker crystallographic alignment and reduced texture intensity. The weaker texture can be attributed to high-temperature-activated grain recovery, which forms more sub-grains, preventing strong grain realignment in a single direction [241,268,273]. As the strain rate at room temperature increases to 2000 s^{-1} , the IPF map in Fig. 9e shows fragmented grains with significant elongation, mainly due to intense strain localization and the reduced time available for grain recovery. The pole figures in Fig. 9g show increased intensity and improved alignment along a

preferred crystallographic direction, consistent with strain-rate-controlled texture strengthening driven by dislocation accumulation [269]. Upon increasing the temperature to 600 °C at a strain rate of 2000 s⁻¹, the IPF maps in Fig. 9f show reduced grain fragmentation and fewer observed internal misorientations than at room temperature. The combination of high strain and high temperature activates cross-slips, leading to accelerated sub-grain formation and rearrangement [268,274,275]. The PF maps in Fig. 9h show weaker texture than in the room-temperature condition, consistent with previous observations for 500 s⁻¹ strain-rate samples.

Fig. 10 presents the evolution of low-angle grain boundaries (LAGB, 2°<θ<15°) and high-angle grain boundaries (HAGB, θ>15°) as a function of strain rate and deformation temperature. The highest fraction of HAGBs is present in the as-sintered condition. This is a characteristic representation of extensive grain-boundary migration and pore elimination at high sintering temperatures, which promotes the formation of strain-free equiaxed grains surrounded by HAGBs. Sintering-induced plastic deformation reduces the dislocation density, yielding a low fraction of substructure grains and, consequently, a low proportion of LAGBs [211,276,277]. Upon increasing the strain rate from 500 s⁻¹ to 2000 s⁻¹, a significant reduction in HAGBs is observed. This is primarily attributed to rapid dislocation and sub-grain structure formation associated with high strain rate [268]. The original HAGB structure in the as-sintered state becomes increasingly fragmented, eventually giving way to LAGBs. A similar reduction in the fraction of HAGBs is observed as temperature increases at the same strain rates. This reduction is mainly due to enhanced dynamic recovery during deformation. As the temperature increases, thermally activated dislocations climb and rearrange, promoting the formation and stabilization of subgrain structure, thereby increasing the fraction of LAGBs [229,268]. A comparable decrease in HAGBs and increase in LAGBs under high-strain-rate loading have also been reported for stainless steels produced by other AM techniques [238,278], confirming that dynamic recovery-driven sub-grain formation is a common deformation mechanism in additively manufactured austenitic stainless steels.

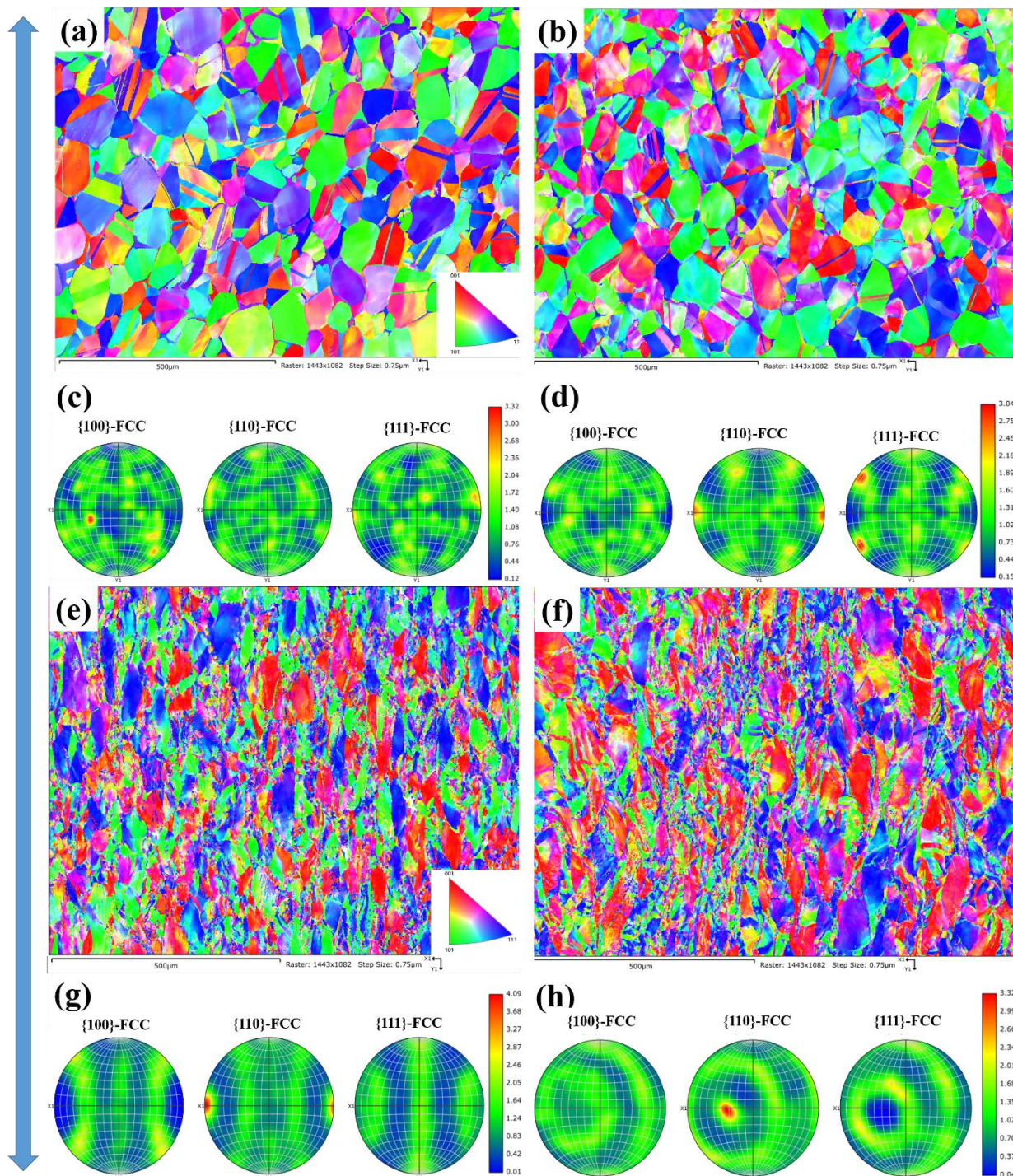


Figure 8-9 Inverse pole figure (IPF) maps and corresponding FCC pole figures showing the effects of strain rate and temperature on microstructural and texture evolution. IPF maps are shown for deformation at 500 s⁻¹ at RT (a) and 600 °C (b), and at 2000 s⁻¹ at RT (e) and 600 °C (f). The corresponding {100}, {110}, and {111} FCC pole figures are presented in (c, d) and (g, h), respectively.

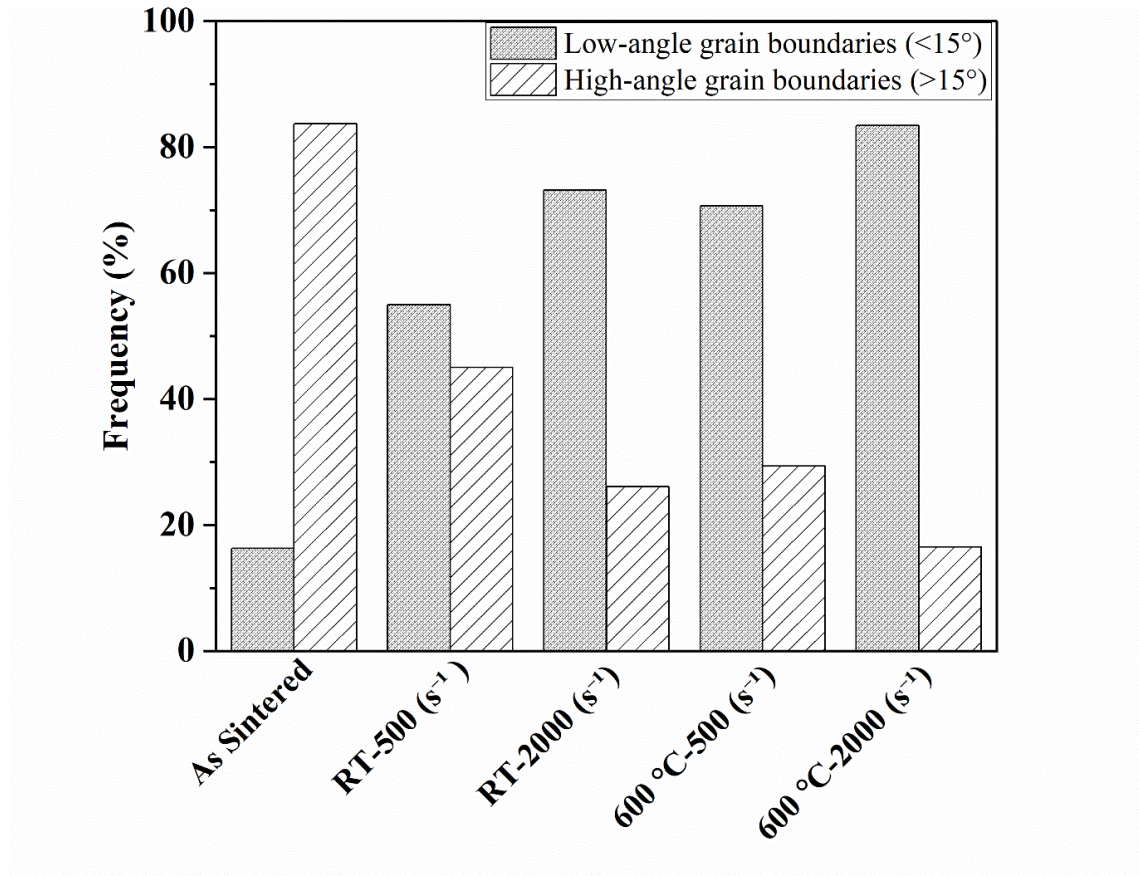


Figure 8-10 Grain boundary angle distribution showing the effect of strain rate and temperature on the fractions of low-angle ($2^\circ < \theta < 15^\circ$) and high-angle ($\theta > 15^\circ$) grain boundaries.

Fig. 11 shows Kernel Average Misorientation (KAM) maps showing the misorientation in the microstructure for different strain rates and temperatures. Increasing the strain rate from 500 s^{-1} (Fig. 11(a)) to 2000 s^{-1} (Fig. 11(b)) at the same temperature (RT) results in a significant increase in misorientation spread, mainly due to higher lattice strain energy, which leads to more dislocations and misorientations [237]. Increasing the temperature from RT to 600°C reduces the overall misorientation due to thermally activated recovery. To further analyze the effect of dynamic compression, the dislocation density for each of the specimens was calculated. Dislocation density represents the amount of lattice defects stored in the material and reflects the level of plastic deformation and strain hardening.

The geometrically necessary dislocation (GND) density was estimated from electron backscatter diffraction (EBSD) data using kernel average misorientation (KAM) maps (Fig. 11), using Equation 6:

$$\rho_{GND} = \frac{K \theta_{KAM}}{b s_r} \quad (6)$$

Where ρ_{GND} is dislocation density, θ_{KAM} is the kernel average misorientation obtained from EBSD analysis, b is the Burgers vector (0.255 nm), s_r is the EBSD step size (0.75 μm), and K is a constant. The results show that at room temperature, increasing the strain rate from 500 s^{-1} to 2000 s^{-1} raises the GND density from 2.3×10^{14} to $3.4 \times 10^{14} \text{ m}^{-2}$, indicating enhanced dislocation accumulation. At 600 $^{\circ}\text{C}$, the GND density decreases to $1.6 \times 10^{14} \text{ m}^{-2}$ at 500 s^{-1} and to $2.7 \times 10^{14} \text{ m}^{-2}$ at 2000 s^{-1} , reflecting thermally activated recovery processes.

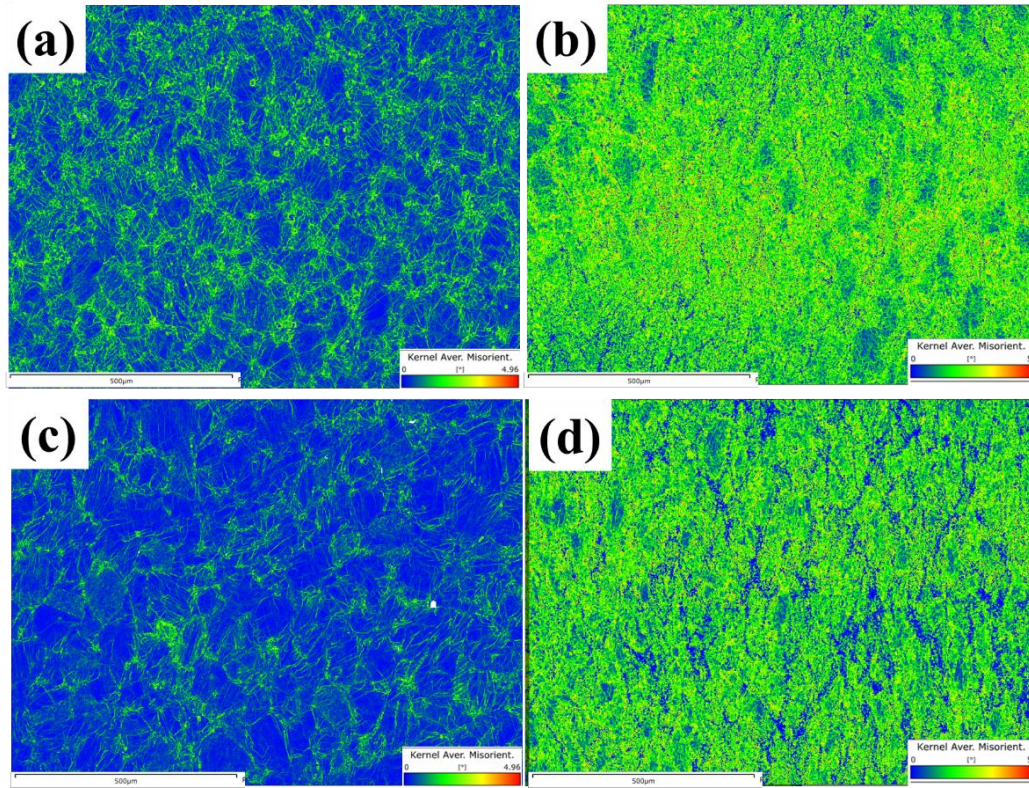


Figure 8-11 Kernel average misorientation (KAM) maps obtained from EBSD analysis after SHPB at: (a) 500 s^{-1} at RT, (b) 2000 s^{-1} at RT, (c) 500 s^{-1} at 600 $^{\circ}\text{C}$, and (d) 2000 s^{-1} at 600 $^{\circ}\text{C}$.

Fig. 12 shows the twin boundary fraction as a function of strain rate and temperature. In the as-sintered state, the specimen shows the highest twin volume fraction, whereas upon high-strain-rate deformation, the proportion decreases significantly. At room temperature, increasing the strain rate results in a significant drop in twin volume percentage from 32% to 2%, indicating dislocation accumulation at the high strain rate that disturbs coherent $\Sigma 3$ annealing twin boundaries [75,76]. A

similar trend is also observed at 600°C, where increasing the strain rate from 500 to 2000 s⁻¹ reduces the twin fraction from 35% to 3%. The cumulative effects of high strain rate and thermally activated dislocations promote the disruption and migration of twin boundaries.

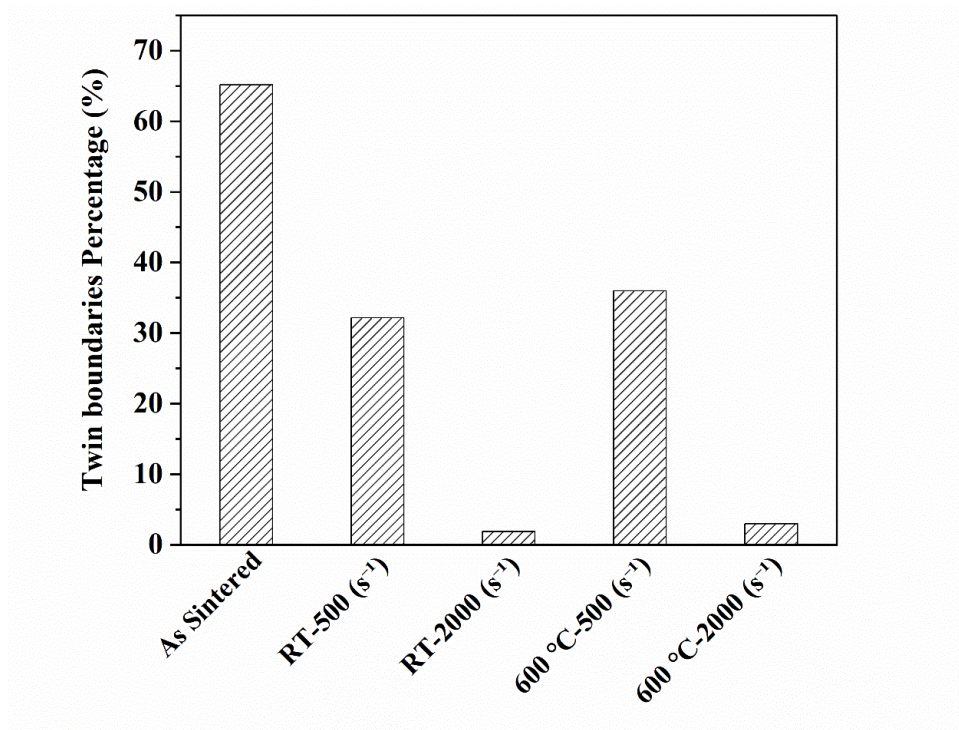


Figure 8-12 Twin volume fraction for the BJ LPBF-cut as-sintered condition and after deformation at 500 s⁻¹ and 2000 s⁻¹ at RT and 600 °C.

8.3.5 Effect of particle size distribution on dynamic response

Fig. 13 (a–c) compares the true stress–true strain responses of binder-jetted 316L stainless steel produced using LPBF-cut (broad PSD) and MIM-cut (narrow PSD) powders over a temperature range from room temperature (RT) to 600 °C and at strain rates of 500, 1000, and 2000 s⁻¹. For both powder conditions, the materials exhibit similar deformation behavior, characterized by rapid initial strain hardening followed by gradual saturation toward peak stress. Between room temperature and 450 °C, the flow curves and peak stresses of the two conditions remain relatively close at each strain rate. At 600 °C, however, a clearer separation is observed, particularly at 2000 s⁻¹, where the MIM-cut samples consistently exhibit lower peak stresses (Fig. 13c).

As shown in Fig. 13(d), the maximum true stress decreases with increasing temperature for both PSDs due to pronounced thermal softening. Although LPBF-cut samples display slightly higher

peak stresses across most temperatures, the difference between the two powder conditions remains modest from room temperature to 450 °C, especially compared with the larger strength differences typically reported under quasi-static loading [264,279,280]. At 600 °C and 2000 s⁻¹, the LPBF-cut samples exceed the MIM-cut samples by approximately 50–60 MPa (≈15–18 %). This indicates that under combined high-temperature and high-strain-rate loading, the influence of powder size distribution becomes more evident, although the overall difference remains moderate.

The similarity in dynamic response between the LPBF-cut and MIM-cut materials indicates that PSD does not play a significant role in controlling strength under SHPB loading conditions by influencing part densification and microstructure (Figs. 3 and 4) [78,79]. SHPB deformation occurs over microsecond time scales, which limits the contribution of pore growth, collapse, or damage accumulation to the overall response. Instead, dynamic strength in austenitic stainless steels is governed primarily by dislocation-controlled mechanisms, including dislocation multiplication, interaction, and limited recovery under high-strain-rate loading [279,280]. Grain structure and associated dislocation accumulation capacity, therefore, play a more dominant role in determining the SHPB response [281]..

The slightly greater divergence observed at 600 °C and 2000 s⁻¹ in Fig. 13(d) can be attributed to the combined effects of thermal softening and high strain-rate hardening. At 600 °C, thermally activated recovery enhances dislocation mobility and reduces the stored dislocation density, leading to lower flow stress [282–285]. At the highest strain rate (2000 s⁻¹), strain-rate hardening intensifies, whereas at lower strain rates (500–1000 s⁻¹) the increase is limited, resulting in a smaller difference in dynamic strength.

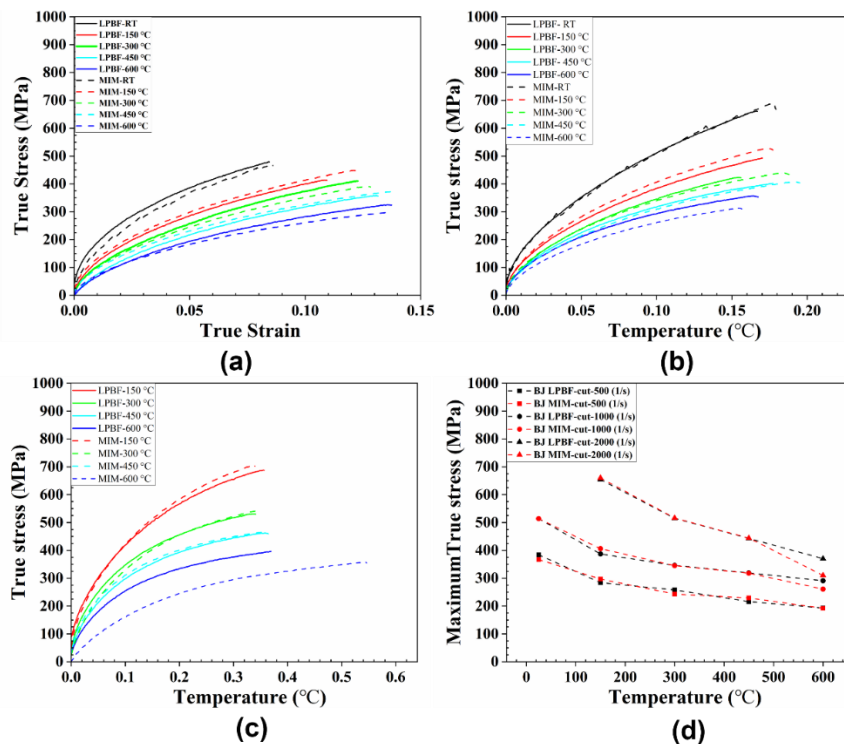


Figure 8-13 (a–c) True stress–true strain responses of BJ-SS 316L comparing LPBF-cut and MIM-cut powders under SHPB test at strain rates of 500, 1000, and 2000 s⁻¹ from RT to 600 °C. (d) Corresponding maximum true stress as a function of temperature. Maximum stresses were extracted at $\varepsilon = 5\%$ (500 s⁻¹), $\varepsilon = 10\%$ (1000 s⁻¹), and $\varepsilon = 30\%$ (2000 s⁻¹).

8.3.6 Effect of HIP on dynamic response

Fig. 14 and 15 present the true stress–true strain responses of BJ LPBF-cut and MIM-cut samples, respectively, in the as-sintered and as-HIPed condition over temperatures ranging from room temperature (25 °C) to 600 °C and at strain rates of 500, 1000, and 2000 s⁻¹. For all cases, the specimens exhibit similar dynamic flow characteristics, with rapid strain hardening at low strain followed by gradual saturation toward peak stress. Increasing temperature produces thermal softening, whereas increasing the strain rate from 1000 to 2000 s⁻¹ increases the flow and peak stresses, confirming clear strain-rate sensitivity. Figs. 14(d, e, f) and 15(d, e, f) show that the maximum true stress is obtained at room temperature, which further decreases with increasing temperature for both powder types and processing conditions. The difference in maximum stress for the as-sintered and as-HIPed conditions remains relatively small across the temperature and strain-rate ranges. In some cases, the as-sintered specimens exhibit slightly higher peak stress,

whereas in others the values are nearly the same as those for as-HIPed, indicating that although HIPing improves densification, it has no effect on improving dynamic compressive strength [263].

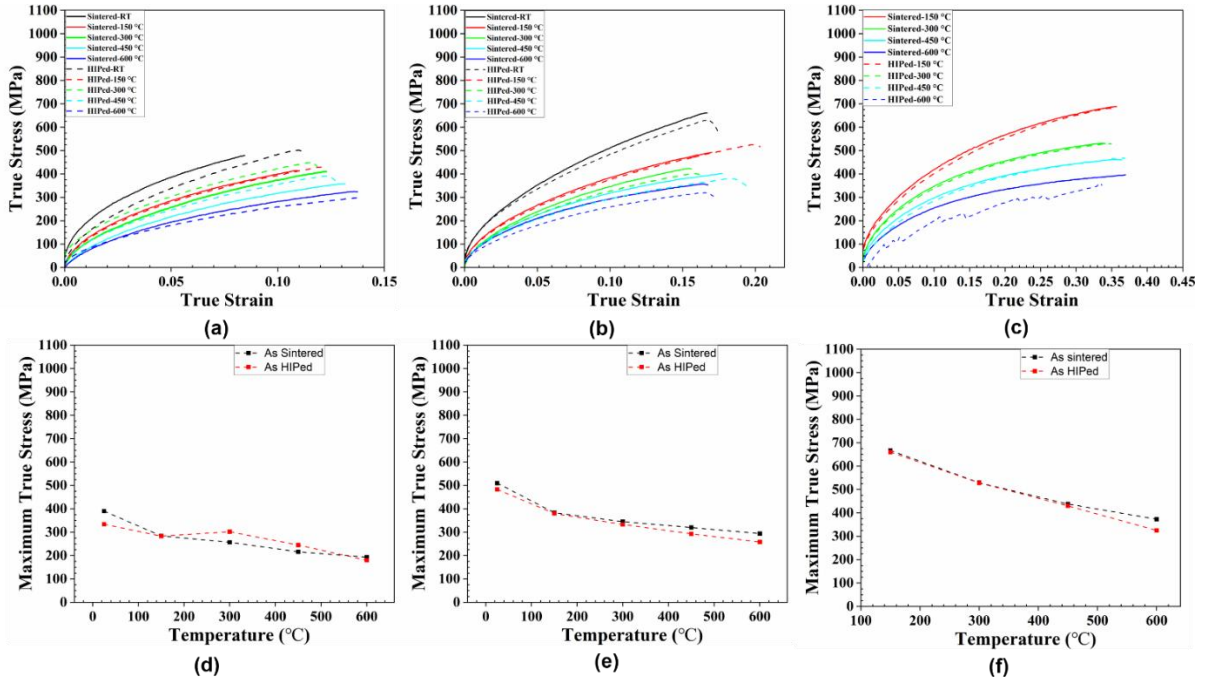


Figure 8-14 (a, b, c) True stress–true strain curves of BJ-SS 316L from LPBF-cut powder in as-sintered and as-HIPed conditions tested from room temperature to 600 °C at strain rates of 500 s⁻¹, 1000 s⁻¹, and 2000 s⁻¹. (d, e, f) Maximum true stress as a function of temperature for the as-sintered and as-HIPed samples. The maximum true stress values were extracted at $\epsilon = 5\%$ for 500 s⁻¹, $\epsilon = 10\%$ for 1000 s⁻¹, and $\epsilon = 30\%$ for 2000 s⁻¹.

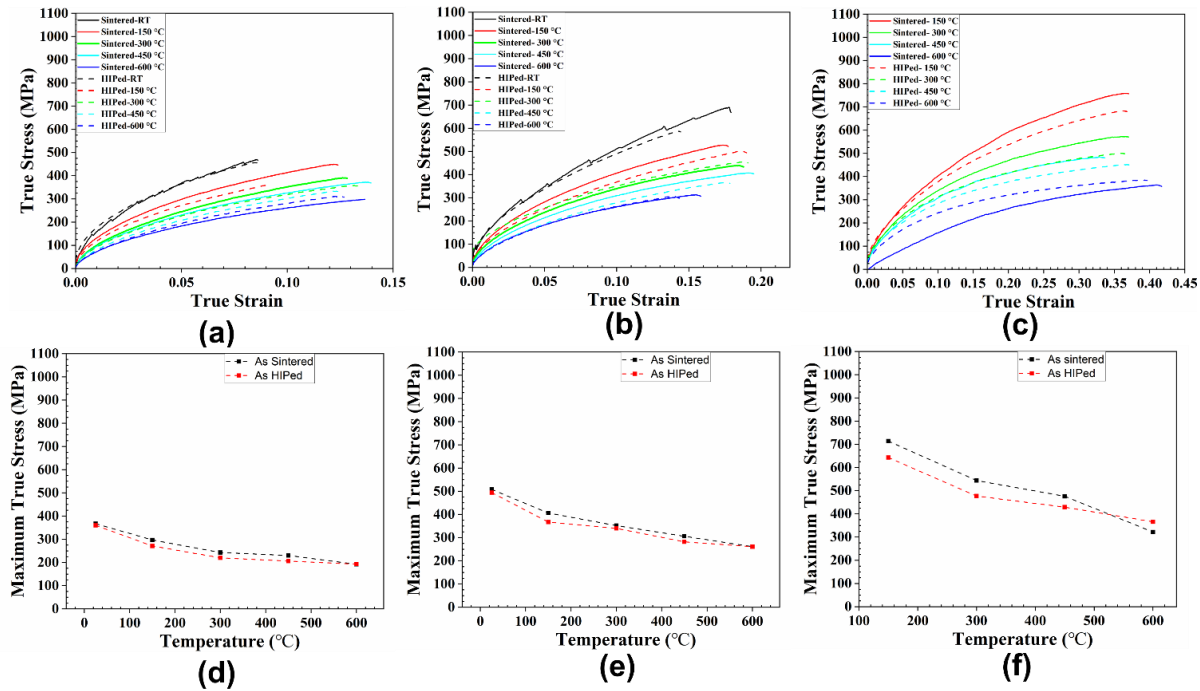


Figure 8-15 True stress–true strain curves of BJ-SS 316L from MIM-cut powder in as-sintered and as-HIPed conditions tested from room temperature to 600 °C at strain rates of 500 s⁻¹, 1000 s⁻¹, and 2000 s⁻¹. (d, e, f) Maximum true stress as a function of temperature for the as-sintered and as-HIPed samples. The maximum true stress values were extracted at $\epsilon = 5\%$ for 500 s⁻¹, $\epsilon = 10\%$ for 1000 s⁻¹, and $\epsilon = 30\%$ for 2000 s⁻¹.

8.3.7 Material constitutive modelling Results

Four constitutive models, Johnson–Cook (JC), modified Johnson–Cook (m-JC), Hensel–Spittel (HS), and modified Hensel–Spittel (m-HS), are evaluated to describe the dependence of flow stress on plastic strain, strain rate, and temperature.

Fig. 16 compares the predictive capabilities of the Johnson-Cook (JC) and modified Johnson-Cook (m-JC) models for flow stress. The JC model (Fig. 16a-c) exhibits limited accuracy at lower strain rates, where it fails to reproduce the experimentally observed stress-strain curvature, particularly at lower temperatures. Although increasing temperature and strain rate improve overall agreement between simulations and experiments, the characteristic curvature of the experimental response is still inadequately captured by the JC formulation. The modified JC model (Fig. 16 d-f) exhibits substantially improved agreement across all strain rates and temperatures. By coupling strain, strain-rate, and temperature effects, the modified formulation accurately reproduces both the shape

and magnitude of the experimental curves, achieving prediction accuracies of approximately 94 % at lower strain rates and limiting the maximum error to about 2-4 % at 2000 s⁻¹.

The limitation of the JC model can be explained by analyzing the change in the strain-hardening exponent (n). Fig. 17 shows the variation in ' n ' with temperature at strain rates of 500, 1000, and 2000 s⁻¹. At strain rates of 500 and 1000 s⁻¹, the work-hardening exponent exhibits only minor variation with temperature, indicating that strain hardening remains relatively stable in the early plastic regime. The JC model treats strain hardening as an uncoupled, temperature-independent term, which limits its ability to capture the experimentally observed curvature and accuracy at lower temperatures and strain rates. At the highest strain rate of 2000 s⁻¹, a pronounced reduction in the work-hardening exponent with increasing temperature is observed, reflecting enhanced thermal softening and stronger coupling between deformation mechanisms. This increased coupling partially explains the improved agreement between the JC predictions and experimental data at high strain rates, although discrepancies in curvature remain.

Fig. 18(a-c) presents the performance of the Hensel Spittel (HS) model. The HS model formulation (Eq.8) provides improved curve-shape fidelity relative to JC-based models due to its intrinsic coupling of strain hardening, strain-rate sensitivity, and thermal softening. This model captures the overall trends in strain rate and temperature, yielding an average prediction accuracy of approximately 96%, although minor deviations persist at higher strain rates. Fig. 18(d-f) presents the performance of the modified Hensel Spittel (m-HS) models. The m-HS model (Fig. 18 d-f) further enhances predictive capability, resulting in near-perfect agreement with the experimental data across all conditions. At the highest strain rate of 2000 s⁻¹, prediction accuracy approaches 98% for all investigated temperatures.

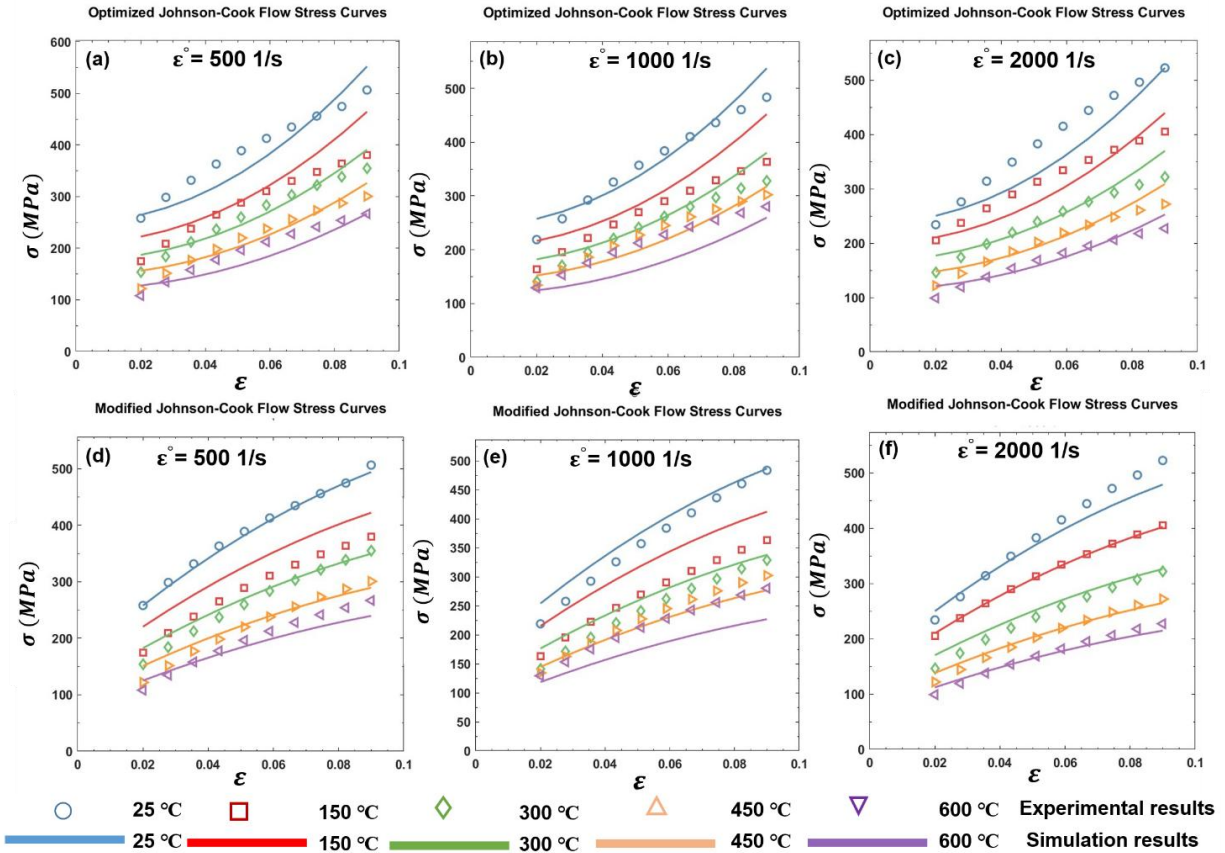


Figure 8-16 Comparison between experimentally measured and model-predicted true stress–true strain curves using the Johnson–Cook (JC) (a-c) and Modified Johnson–Cook (m-JC) (d-f) constitutive models for BJ-SS 316L specimens at strain rates of 500 s^{-1} , 1000 s^{-1} , and 2000 s^{-1} .

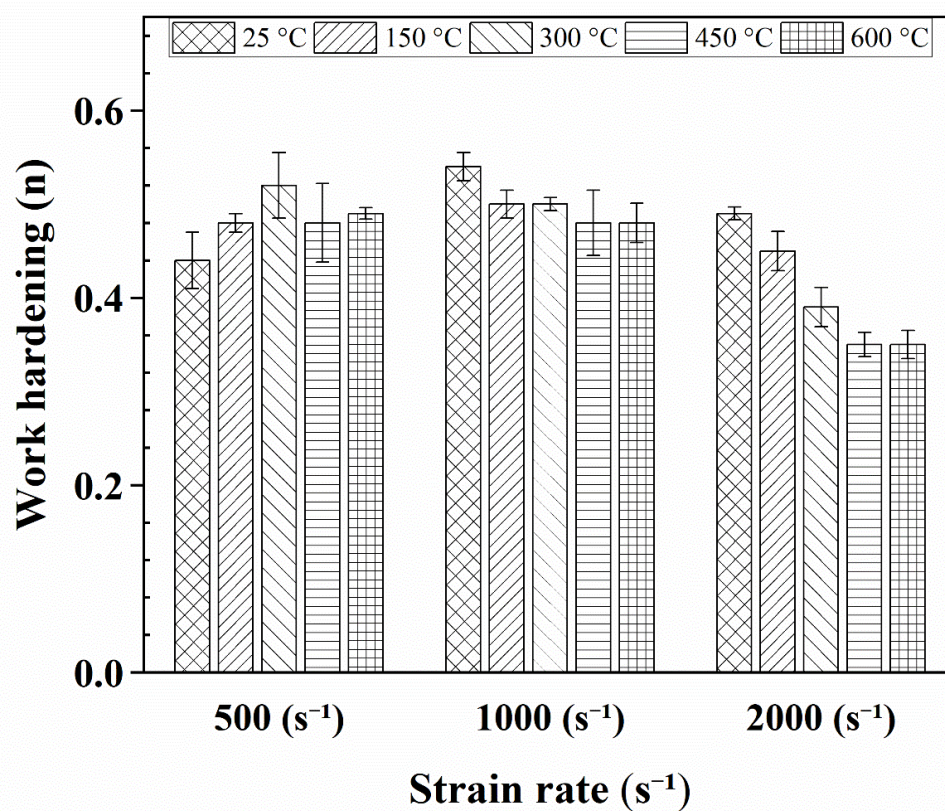


Figure 8-17 Work-hardening exponent (n) as a function of strain rate (500, 1000, and 2000 s^{-1}) at different test temperatures (25–600 °C) for as-sintered BJ SS 316L.

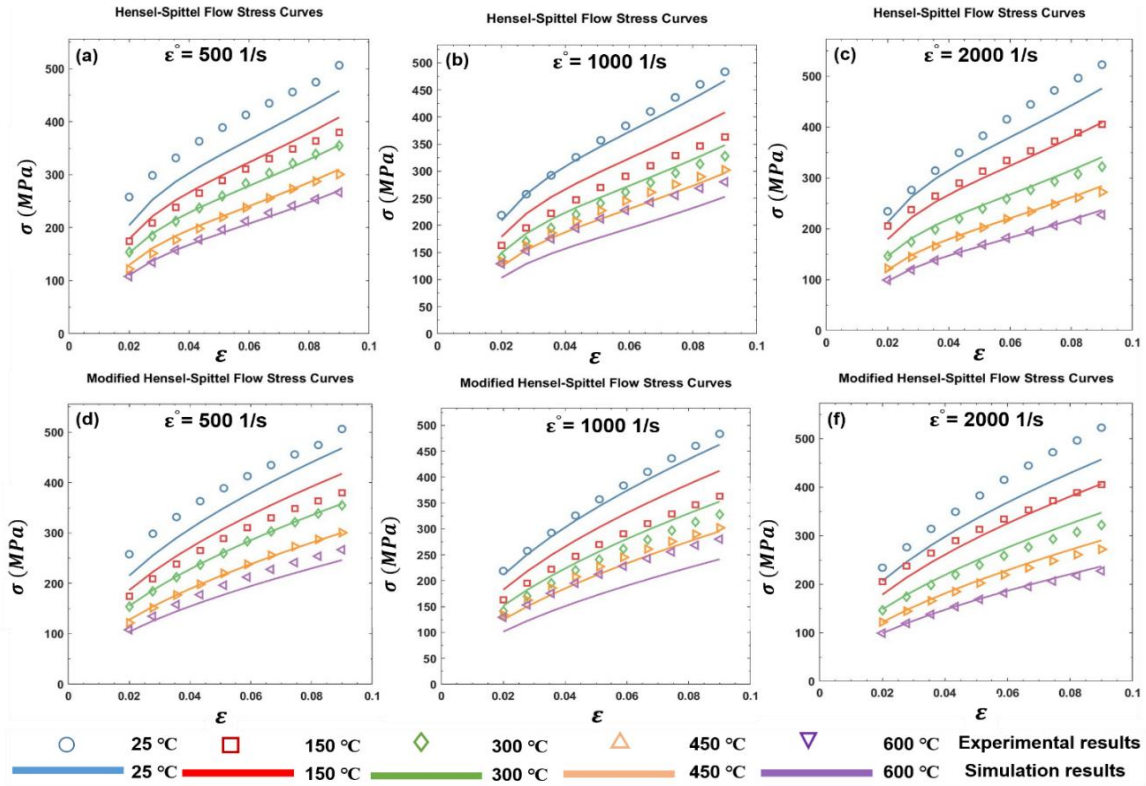


Figure 8-18 Comparison between experimentally measured and model-predicted true stress–true strain curves using the Hensel-Spittel (HS) (a-c) and Modified Hensel-Spittel (MHS) (d-f) constitutive models for BJ SS 316L at strain rates of 500 s⁻¹, 1000 s⁻¹, and 2000 s⁻¹.

Fig. 19 presents contour maps of the Average Absolute Relative Error (AARE) as a function of temperature and strain rate for the four constitutive models. The contour lines represent iso-error levels, while the color gradients indicate the magnitude of normalized prediction deviation, with lower values corresponding to higher predictive accuracy. Distinct error localization patterns are observed for each model. The optimized JC model exhibits pronounced high-error regions, particularly at intermediate temperatures, revealing its limited ability to simultaneously capture thermal softening and strain-rate hardening. Although the m-JC formulation reduces peak error magnitudes, localized high-error bands persist, indicating only partial improvement in thermo-viscoplastic coupling. The HS model generates a smoother and more gradually varying error field, reflecting improved sensitivity to exponential temperature and strain-rate effects. The m-HS model shows the most uniform and lowest-error distribution across the entire domain, with minimal localized peaks even under high strain-rate and elevated-temperature conditions. Overall, the

contour topology demonstrates that strong thermo-strain-rate coupling is essential for robust prediction of the dynamic deformation response of BJ-316L parts.

A quantitative comparison of model performance is summarized in Table 4, where the m-HS model exhibits the highest coefficient of determination ($R^2 = 0.951$) and the lowest average absolute relative error (AARE = 5.49%).

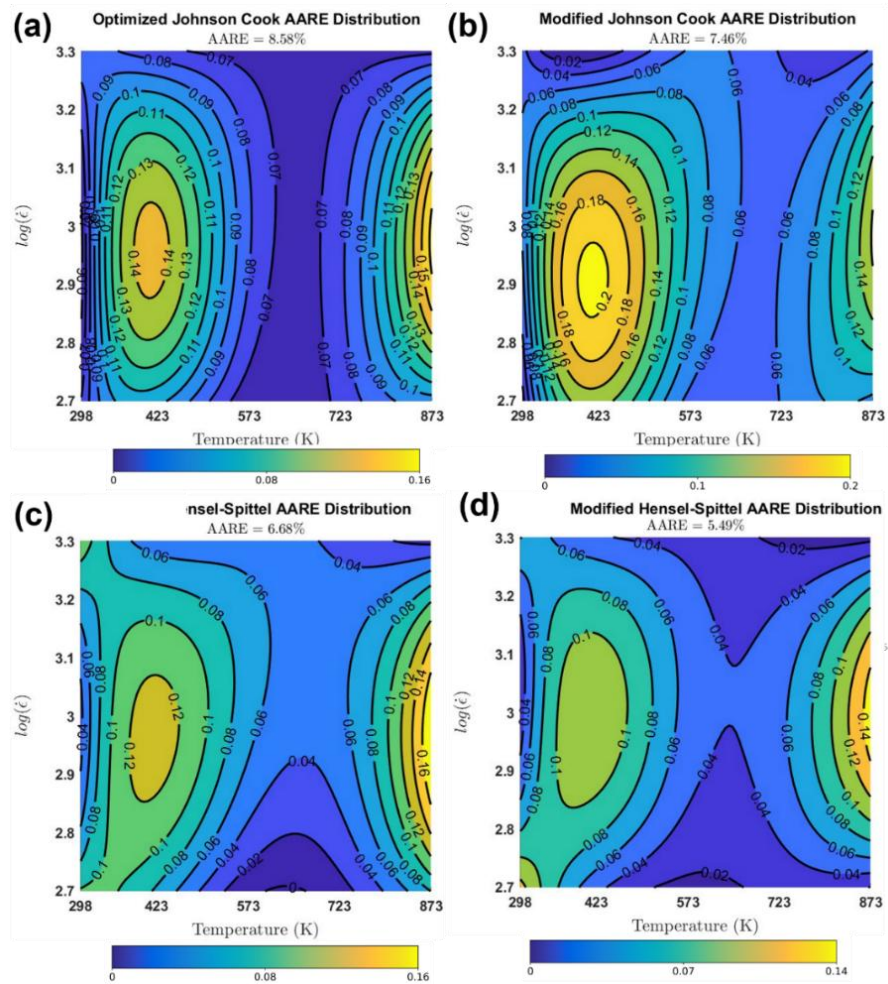


Figure 8-19 Contour maps of the Average Absolute Relative Error (AARE) as a function of temperature and strain rate for different constitutive models: (a) optimized Johnson–Cook, (b) modified Johnson–Cook, (c) Hensel–Spittel, and (d) modified Hensel–Spittel.

Table 8.4 Statistical comparison of constitutive model performance, using the coefficient of determination (R^2) and the % average absolute relative error (AARE).

Models/Parameters	JC	m-JC	HS	m-HS
R^2	0.915	0.9254	0.933	0.951
AARE (%)	8.58	7.46	6.68	5.49

8.4 Conclusions

This study evaluated the high-strain-rate compressive behavior of binder-jetted (BJ) 316L stainless steel by examining the combined influence of strain rate (500, 1000, 2000 s^{-1}), temperature (25, 150, 300, 450, 600 $^{\circ}C$), powder size distribution (PSD), and hot isostatic pressing (HIP). Based on the experimental observations and constitutive modeling results, the following conclusions are drawn:

- 1- The dynamic mechanical response of BJ 316L is primarily governed by the interplay between strain-rate hardening and thermal softening. Increasing the strain rate from 500 to 2000 s^{-1} increases the peak stress by approximately 57%, whereas increasing the temperature from room temperature to 600 $^{\circ}C$ reduces the strength by 60%. Temperature exerts a stronger influence than strain rate, confirming that thermally activated dislocation motion and dynamic recovery dominate deformation at elevated temperatures.
- 2- BJ specimens exhibit slightly lower peak stresses (10%) than wrought 316L due to residual porosity and microstructural heterogeneity. However, both materials display comparable strain-rate hardening and similar flow-curve characteristics. This indicates that deformation mechanisms in both cases are governed by dislocation-controlled plasticity rather than by the processing route, highlighting the viability of BJ for crash-relevant applications.
- 3- The BJ-316L microstructure is significantly impacted by high-strain-rate compression test parameters. Increasing strain rate increases dislocation density, misorientation, and

proportion of LAGBs, and reduces the annealing twin boundaries fraction. However, increasing temperature reduces dislocation density and the LAGB fraction due to dynamic recovery.

- 4- PSD has minimal impact on the high-strain-rate compressive behavior of BJ parts. This is because under microsecond SHPB loading, dynamic strength is governed mainly by dislocation kinetics rather than modest variations in porosity or initial packing density.
- 5- HIP does not substantially enhance dynamic compressive strength. The minimal difference between as-sintered and HIPed conditions confirms that high-strain-rate strength is controlled predominantly by intrinsic rate- and temperature-dependent slip mechanisms rather than by small residual pores addressed by sintering and HIP.
- 6- The modified Hensel–Spittel (m-HS) model, having strong intrinsic thermo-viscoplastic coupling, demonstrated the highest predictive capability ($R^2 \approx 0.95$; lowest AARE), outperforming the Johnson–Cook and modified Johnson–Cook models. M-HS provides a reliable route to simulate the deformation behavior of BJ-316L in crash-relevant loading conditions

Acknowledgments

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CHAPTER 9 CONCLUSION AND RECOMMENDATIONS

9.1 Summary of work

This research is organized into three main parts, each targeting a key challenge in binder-jetted stainless steel components. First, anisotropic sintering behavior is investigated by evaluating the effects of sintering kinetics, viscosity, temperature, printing orientation, and microstructural evolution on the shrinkage and deformation behavior of 316 L and 17-4 PH stainless steels. Then, the role of powder size distribution on densification kinetics, shrinkage magnitude, anisotropic sintering behavior, and microstructural evolution was examined using MIM-cut and LPBF-cut powders. Finally, the high-strain-rate compressive behavior of binder-jetted stainless steels was evaluated under crash-relevant loading conditions, accounting for temperature, strain rate, powder size distribution, and hot isostatic pressing. The main conclusions resulting from the analysis of these investigations are summarized as follows:

- Binder-jetted stainless steels exhibit pronounced anisotropic shrinkage during sintering, characterized by quasi-isotropic deformation within the powder bed plane and significantly larger shrinkage along the build direction. This anisotropy is primarily attributed to the layer-by-layer printing architecture and to the collapse of the primitive lines formed during powder deposition, both of which directly affect dimensional accuracy.
- Sintering temperature governs both densification kinetics and anisotropic shrinkage in binder-jetted stainless steels. Increasing temperature induces a transition from grain-boundary diffusion to volume diffusion, thereby significantly accelerating shrinkage, grain growth, and densification. Moreover, higher temperatures intensify anisotropic shrinkage by promoting the collapse of directionally aligned pores, particularly primitive lines along the build direction, thereby increasing Z-axis (build direction) contraction. However, at elevated temperatures, progressive pore homogenization reduces directional differences, stabilizing the anisotropy sintering ratio (ASR) despite continued overall shrinkage.
- Binder jetting (BJ) produces comparable green density, porosity distribution, and primitive line features in both 316L and 17-4 PH stainless steels. However, despite these similar initial conditions, the two alloys exhibit markedly different anisotropic shrinkage behavior and sintering kinetics. The diffusion-mechanism transition occurs at a lower temperature in 17-4 PH (≈ 1250 °C) than in 316L (≈ 1300 °C). Moreover, the activation energies for both grain-boundary and volume

diffusion are lower in 17-4 PH (approximately 100–125 kJ/mol and 250–320 kJ/mol, respectively) compared to 316L (25–162 kJ/mol and 490–623 kJ/mol, respectively). These differences arise primarily from phase constitution and alloy composition. During high-temperature sintering, 316L remains predominantly austenitic and forms only about 6–8% δ -ferrite, whereas 17-4 PH develops a significantly higher δ -ferrite fraction (approximately 20–30%). The increased δ -ferrite content in 17-4 PH enhances atomic diffusivity, thereby accelerating densification, increasing total shrinkage, and intensifying anisotropic deformation compared to 316L.

- In binder-jetted stainless steel, anisotropic sintering behavior is strongly influenced by temperature- and porosity-dependent viscous modulus properties. Directional pore architecture results in higher bulk and shear viscosities along the build direction at lower temperatures, creating anisotropic resistance to densification. As temperature increases and the mechanism transitions to volume diffusion, viscosity decreases due to enhanced atomic mobility, accelerating pore collapse, and intensifying Z-direction shrinkage. At higher temperatures, pore homogenization reduces directional viscosity differences, leading to a more isotropic viscous response.
- Anisotropic sintering in both binder-jetted 316L and 17-4 PH primarily affects dimensional shrinkage rather than final microstructure. Although the build direction exhibits greater contraction due to the collapse of aligned pores and primitive lines, grain size, phase distribution, and final density remain largely independent of printing orientation. The activation energy associated with grain boundary and volume diffusion is also consistent across all print axes, confirming that the underlying diffusion mechanisms are orientation-independent. In 316L, δ -ferrite fraction and grain growth evolve similarly in all directions, while in 17-4 PH, despite stronger anisotropic shrinkage, the martensitic matrix and higher δ -ferrite content develop uniformly. Thus, anisotropic deformation reflects directional pore collapse and viscous response rather than intrinsic microstructural anisotropy.
- Powder size distribution (PSD) significantly influences densification behavior and dimensional stability during sintering in binder-jetted 316L stainless steel. Narrower PSD (MIM-cut) promotes faster densification and higher final density due to enhanced surface area and smaller pore size, but results in greater total shrinkage and stronger anisotropic shrinkage (40%). In contrast, broader PSD (LPBF-cut) improves green packing and reduces anisotropic shrinkage (20%), although it leads to slightly lower final densification.

- The mechanical response of binder-jetted 316L stainless steel is strongly influenced by both strain rate and temperature. Increasing strain rate from 500 to 2000 s⁻¹ enhances peak stress by approximately 25-30%, while increasing temperature from room temperature to 600 °C reduces strength by about 35–40% due to thermal softening. Although binder-jetted material exhibits approximately 10–20% lower absolute strength than wrought 316L, it demonstrates similar strain-rate sensitivity, deformation stability, and temperature dependence. These results indicate that binder jetting is a promising manufacturing route for crash-relevant applications, provided that density and microstructural integrity are properly controlled.
- The microstructural evolution of binder-jetted 316L under high-strain-rate loading is controlled by the coupled influence of strain rate and temperature. Increasing strain rate promotes dislocation multiplication, subgrain formation, and higher fractions of low-angle grain boundaries, thereby contributing to strain hardening. In contrast, increasing temperature enhances dynamic recovery, reduces stored dislocation density, and lowers work-hardening capacity. Due to the high stacking-fault energy of 316L and the short deformation time associated with high-strain-rate testing, deformation is primarily dominated by dislocation slip and recovery rather than extensive dynamic recrystallization. Overall, the balance between strain-rate-induced hardening and temperature-driven recovery determines the material's dynamic mechanical response.
- Constitutive models, including the Johnson–Cook and Hensel–Spittel formulations and their modified versions, were calibrated to describe the deformation behavior of binder-jetted 316L over a range of high strain rates and temperatures. Accurate prediction requires strong coupling between strain, strain rate, and temperature. The modified Hensel–Spittel model achieved the highest accuracy, whereas the classical Johnson–Cook model underestimated stress–strain curvature at lower strain rates due to its uncoupled formulation. These findings highlight the importance of incorporating thermo-mechanical coupling for reliable modeling of binder-jetted stainless steels under crash-relevant loading conditions.

The results of this study provide a comprehensive framework linking binder jetting process parameters, anisotropic sintering behavior, microstructural evolution, and high-strain-rate mechanical response of stainless steels. The conclusions offer practical guidelines for the design and qualification of binder-jetted stainless steel components for industrial applications.

9.2 Limitations

This thesis focused on the fundamental understanding of anisotropic sintering behavior and high-strain-rate mechanical response of binder-jetted stainless steels. However, given that the investigations were conducted on laboratory-scale specimens and specific processing conditions, the following limitations are identified:

- Complex component geometries may lead to different shrinkage constraints and local stress states during sintering due to thermal gradient. The present study does not allow direct correlation between complex part geometry, local stress, and anisotropic shrinkage behavior. The conclusions are therefore limited to relatively simple geometries such as cubic and cylindrical specimens.
- The sintering viscosity and its anisotropy were not measured experimentally but inferred through model calibration. As a result, the identified bulk and shear viscosity parameters are effective quantities, whose uniqueness and physical validation cannot be fully established independently of the modeling assumptions.
- High-strain-rate mechanical characterization was restricted to uniaxial compressive loading using Split Hopkinson Pressure Bar testing. Real automotive crash scenarios may involve multiaxial stress states, tensile loading, bending, and complex strain paths, which were not addressed in this study.
- The conclusions regarding sintering behavior and dynamic response are limited to binder-jetted 316L and 17-4 PH stainless steels, and their transferability to other alloy systems or binder-jetting feedstocks requires further investigation.

9.3 Future research

In the previous section, the applicability of the proposed framework to complex geometries and industrial-scale components was identified as a current limitation for the widespread adoption of binder jetting in automotive applications. Future research directions are proposed to address these limitations.

- Future work should investigate anisotropic sintering behavior in geometrically complex components, where geometry-dependent shrinkage constraints and thermally induced local

stress states become significant. This should involve controlled experiments on non-prismatic specimens combined with advanced numerical simulations to establish a direct correlation among complex part geometry, thermal gradients, and anisotropic shrinkage behavior. Such studies would clarify the influence of geometry on densification and stress development and extend the applicability of the present conclusions beyond simple cubic and cylindrical laboratory-scale specimens.

- To improve the physical grounding of the sintering model, direct experimental measurement of sintering viscosity and its anisotropy should be pursued. Approaches such as sintering creep experiments under applied load or combined mechanical–thermal testing during sintering would allow independent identification of bulk and shear viscosity parameters and reduce reliance on inverse calibration from shrinkage data.
- Further high-strain-rate mechanical testing under multiaxial loading, tensile impact, and bending conditions is necessary to characterize crash-relevant behavior fully. Extending the investigation to damage tolerance, fracture, and failure mechanisms would improve the reliability of binder-jetted components in safety-critical applications.
- Expanding the experimental and modeling framework to additional stainless steel grades and alloy systems would allow assessment of the generality of the observed trends and support the development of transferable sintering and constitutive modeling strategies for binder jet additive manufacturing.

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