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Evolution of grain boundary network topology in 316L austenitic stainless steel during powder hot isostatic pressing

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ABSTRACT

The grain boundary network evolution of 316L austenitic steel powder during its densification by hot isostatic pressing (HIPing) was investigated. While the as-received powder contained a network of random high angle grain boundaries, the fully consolidated specimen had a large fraction of annealing twins, indicating that during densification, the microstructure evolves via recrystallization. By interrupting the HIPing process at different points in time, microstructural changes were tracked quantitatively at every stage using twin boundary fractions, distribution of different types of triple junctions, and the parameters associated with twin related domains (TRDs). Results revealed that, with increase in temperature, (i) the fraction of annealing twins increased steadily, but they mostly were not part of the grain boundary network in the fully consolidated specimen and (ii) the average number of grains within a TRD, the length of longest chain, and twinning polysynthetic during HIPing and (iii) the powder characteristics and the HIPing parameters have a strong influence on the development of grain boundary network. Based on the results obtained, possible alterations to the HIPing process are discussed, which could potentially allow twin induced grain boundary engineering.

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1. Introduction

Powder hot isostatic pressing (HIPing) is a net shape manufacturing process that is used to produce fully dense components through the application of pressure (P) and temperature (T) on a powder compact for certain amount of time (t), which results in its complete consolidation [1]. Powder HIPed components are currently being used in several industries, including oil and gas, automotive, and aerospace. HIPing is also used to remove residual porosity in castings [1]. Advantages of powder HIPing include better chemical homogeneity, fine grain size, isotropic properties, increased materials utilization, and the ability to produce complex near net shaped components. Additionally, reduced lead time for manufacturing big near net shaped components and ease of in-service inspectability are other important advantages of HIPing.

HIPing, along with other powder based manufacturing processes such as additive manufacturing, is being considered as a potential alternative for producing nuclear reactor components [2,3]. It has been demonstrated that HIPing, owing to the advantages it offers over conventional processing, is a viable manufacturing process for producing pressure retaining components made of 316L for nuclear reactors [4–6]. 316L components produced from rolling and forging are usually used in the solution annealed, recrystallized state. Annealing twins, which are a key microstructural feature of recrystallized 316L austenitic stainless steels, are also observed in the microstructure of powder HIPed specimens (see for e.g., [7,8]).

The presence of a large fraction of annealing twins in the fully consolidated microstructure indicates possibilities to optimize the HIPing process to enhance their fraction in the microstructure (i.e., twin induced grain boundary engineering). The importance of grain boundaries in influencing material properties has long been recognized [9–13]. Specifically, for face centered cubic (FCC) materials which profusely twin, previous studies have shown that twin boundaries, i.e., \( \Sigma 3 \) boundaries in coincidence site lattice
(CSL) framework [14], are resistant towards carbide precipitation [15,16], intergranular stress corrosion cracking (IGSCC) [17–19] and have reduced susceptibility to intergranular hydrogen embrittlement [20].

Grain boundary networks in multiple-twinned materials have previously been studied extensively within the context of grain boundary engineering and control (e.g., see Refs. [19,21–27]). It was shown that, due to their contrasting properties, the response of a material to various intergranular phenomena (e.g., intergranular stress corrosion cracking) is affected not only by the types of grain boundaries present, but also by the way they are interconnected. Since the grain boundary network topology is constrained by the crystallography at the triple junctions, it was suggested that the grain boundary connectivity, apart from quantifying the special boundary fraction (i.e., boundaries with CSL ≤ 29), can be better understood by quantifying the types of triple junctions present [22,24,25,28,29]. Specifically, based on the types of boundaries present at a triple junction (CSL and random), it can either allow a crack to propagate further or act as arresting point. In this approach, the response of grain boundary network towards intergranular phenomena is treated as a correlated percolation problem.

It has previously been demonstrated that in materials that are susceptible to annealing twinning, the recrystallized microstructure consists of multiple-twinned clusters, called twin related domains (TRDs) [23,30–35]. Gertsman [23] noted that the entire microstructure is made up of TRDs and because every cluster originates from one orientation, they are linked to recrystallization. In a TRD, twinning process can proceed to any order, and thus contains twin chains. Therefore, all grains within a TRD are connected by chains of Σ 3 boundaries and are related by Σ 3n misorientations while the outer boundaries of TRDs have crystallographically random orientations. Since any crack propagation will only be through outer boundaries of the TRDs, they represent blocks that are generally immune to percolative phenomena and it was suggested that TRD size could be considered as the characteristic microstructural dimension [23,33]. So, from the point of view of enhanced resistance towards intergranular phenomena, the microstructure must contain large TRDs with multiple twins rather than just annealing twins. Such a microstructure can be achieved by thermomechanical processing, like sequential strain-annealing or one-step strain-annealing [22,36,37].

Reed [30,38] and Cayron [31,33] developed the theory for quantifying multiple twins and identifying TRDs, while Cayron [33] suggested more advanced parameters to quantify multiple twinning, like the averages of number of grains per TRD (\(\langle N_3 \rangle\)), length of longest chain (\(\langle LCC \rangle\)), polysynthetic (\(\langle p \rangle\)), and twinning anisotropy (\(\langle \alpha \rangle\)). For a reconstructed TRD, LCC refers to the largest misorientation between two grains, and is represented by \(n\) in \(\sum 3^n\) notation. In other words, it represents twinning order of the TRD. Polysynthetic quantifies how frequently the individual orientations occur in a TRD. Detailed theory and the procedure for identifying TRDs have previously been reported [30,31,33].

Considering the importance of grain boundaries/grain boundary network in influencing the material performance, an improved understanding of the HIPing process from the standpoint of grain boundary control assumes practical significance. It is, therefore, important to identify the mechanisms/process variables that influence the microstructural development during HIPing, so that they can be controlled (e.g., by altering the HIPing cycle) to produce a desired network of grain boundaries.

Hence, the objective of the present study is two fold. First, to understand the grain boundary network evolution in 316L during HIPing and identify the mechanisms/process variables that influence it. Second, since 316L can be subjected to twin induced grain boundary engineering using thermomechanical processing, and recognizing the fact that HIPing is one such process, to explore possibilities of performing grain boundary engineering during HIPing. So, the data analysis was oriented towards understanding the grain boundary network evolution and quantification of multiple twinning during HIPing. Analysis was performed on specimens that were produced by interrupting the standard HIPing cycle at various stages. In other words, evolutionary microstructural states during HIPing were captured for the analysis.

2. Materials and methods

2.1. Experimental

HIP specimens were produced from nitrogen atomized 316L powder with the chemical composition shown in Table 1 and with a less than 500 μm particle size and a mean size of 80 μm; the particle size distribution is shown in Fig. 1a. The HIPing process consisted of the following steps: the powder was first filled in mild steel canisters of 25 mm diameter, 30 mm height and 2 mm thickness, vibrated and vacuum degassed at room temperature. The canisters were then sealed by hot crimping the evacuation tube. The HIPing cycle consisted of simultaneous application of temperature and pressure at 0.5 °C/min and 0.5 MPa/min, respectively. HIPing was performed at 950 °C, 1000 °C, 1050 °C, and 1120 °C at 103 MPa, without any hold time at those temperatures. Specifically, the HIPing cycle was interrupted by ramping down the temperature and pressure as soon as they reached the set points. A typical HIPing cycle is shown in Fig. 1b. Additionally, one canister (70 mm diameter and 200 mm height) was HIPed at 1160 °C, 103 MPa and held for 4 h (i.e., to full HIP cycle). This sample was then solution annealed at 1050 °C for 1 h and water quenched. All specimens were sectioned, ground and polished using standard metallographic procedures. Final polishing was performed on a vibratory polisher using colloidal silica solution for 2 h. Electron backscatter diffraction (EBSD) maps were acquired on a Field Emission Gun Scanning Electron Microscope (FEG SEM, model: CamScan Maxim), equipped with Aztec EBSD system and a Nordlys II camera. Data were acquired at 20 kV with 0.5 μm step size for the partially consolidated HIPed specimens and 1 μm for the fully consolidated specimen. EBSD maps from 5 randomly selected regions per specimen were acquired for statistical analysis of the data.

2.2. Data analysis

To study the evolution of grain boundary network and the microstructure of 316L powder compact during its densification by HIPing, data have been analyzed by following (i) the evolution of CSL boundaries and types of triple junctions and (ii) the parameters associated with twin related domains. Therefore, in the present study, the fraction of CSL boundaries and triple junction distributions were extracted from EBSD data using MTEX, a MATLAB based open source software [39]. Boundaries with CSL ≤ 29 (with a tolerance angle of 3° from ideal misorientation) were quantified by their length (\(L_3\)) and number (\(N_3\)) fractions. It is pointed out that the quantification was performed for a comparison with those published in the literature and not because all CSL boundaries ≤ 29 contain special properties (except twin boundaries). Following Kumar et al. [22], triple junctions were classified as \(J_0\), \(J_1\), \(J_2\), and \(J_3\) where \(J_0\) constitutes a triple junction with i CSL and (3-i) random boundaries, respectively. Only \(\sum 3, \sum 9, \sum 9\) and \(\sum 27\) boundaries were considered as CSL boundaries in the triple junction analysis.

Since TRDs contain information pertaining to the microstructural development, they were analyzed in detail using ARPGE, a python based software developed by Cayron [33,40]. A caveat
needs to be mentioned regarding the experimental conditions; due to the difficulty in rapidly cooling the samples from the HIPing temperature ($\sim 550^\circ$C/min), the data reported do not necessarily correspond to the actual high temperature state of the sample. Nevertheless, the trends observed in the data from the samples HIPed at different temperatures, as will be shown below, still provide valuable information on the microstructural evolution.

3. Results

The grain boundary misorientation maps of three as-received powder particles of \( \sim 35 \mu m, 90 \mu m, \) and \( 225 \mu m \) size and with an average grain size of \( 6 \mu m, 10 \mu m, \) and \( 14 \mu m \), respectively, are shown in Fig. 2. Here, the grain boundary misorientations are represented according to the colouring scheme proposed by Patala et al. [41,42]. This colouring scheme allows the representation of complete misorientation information (angle and axis) of the grain boundaries using the legend shown in Fig. 2e. In other words, each boundary is uniquely coloured based on its misorientation angle and axis, without broadly classifying it to be part of one category or the other (e.g., high angle and low angle, and CSL and random). For a comparison, the particle in Fig. 2c is shown with only \( \Sigma 3, \Sigma 9, \) and \( \Sigma 27 \) boundaries highlighted in Fig. 2d. The rapidly solidified powders predominantly contain high angle grain boundaries, but few low angle grain boundaries are also seen (i.e., boundaries with misorientation \( < 5^\circ \), which are coloured in white in Fig. 2b). Moreover, the \( \Sigma 3 \) grain boundaries in all particles are not long and straight, but have appearance similar to any other high angle grain boundary. In Fig. 3, a representative grain boundary misorientation map of the fully consolidated specimen is shown. It is seen that annealing twins (i.e., \( \Sigma 3 \) boundaries in the coincidence site lattice framework [14]) form a significant fraction of the grain boundaries present in the microstructure. The appearance of parallel sided \( \Sigma 3 \) boundaries, i.e., annealing twins, in the orientation map suggests that they had formed as a result of recrystallization during HIPing.

The fractions of \( \Sigma 3^n \) boundaries (up to \( n = 2 \)) and triple junctions in the as-received powder are shown in Table 2; also shown are the statistics for the fully consolidated specimen for comparison. In the powder, as expected, most of the triple junctions contain random boundaries with \( J_0 \) fraction being highest. The statistics for the fully consolidated specimen, on the other hand, show an increased fraction of \( \Sigma 3^n \) boundaries. However, most of the \( \Sigma 3 \) boundaries are part of \( J_1 \). Comparing the microstructures and the statistics for the powder and the fully consolidated specimen (Figs. 2 and 3, and Table 2), it is clear that the microstructure changed from the one containing random boundaries in the as-received powder to a twin dominated one in the fully consolidated specimen. In order to understand this change, microstructures representative of those present at various stages during HIPing were analyzed.

Representative grain boundary misorientation maps of partially consolidated HIPed samples are shown in Fig. 4. Extensive formation of subgrain boundaries, as a consequence of the incipient deformation of the powder particles, is seen in the sample HIPed at \( 950^\circ \)C (Fig. 4a). Along with porosity, powder particles are also clearly seen because of incomplete consolidation. In the sample HIPed at \( 1000^\circ \)C, incipient formation of annealing twins (quantitatively, seen as parallel sided boundaries; also see the legend for \( \Sigma 3 \) boundary in Fig. 4e) is seen along with the presence of fewer subgrain boundaries (Fig. 4b) as compared to the sample HIPed at \( 950^\circ \)C. A decrease in the fraction of subgrain boundaries along with concomitant increase in the fraction of annealing twins is seen in the sample HIPed at \( 1050^\circ \)C (Fig. 4c). Further increase in the annealing twin fraction and a decrease in subgrain boundaries is seen in the sample HIPed at \( 1120^\circ \)C (Fig. 4d). Moreover, in certain regions of the partially consolidated samples, it was observed that smaller particles deformed more than larger particles; a representative misorientation map is shown in Fig. 5. Here, it is seen that annealing twins have formed profusely in smaller particles that are decorated around larger, non-deformed particles. It is also seen that the non-deformed particles have retained their identity (i.e., shape and grain boundary characteristics) of the as-received state (see Fig. 2).

Annealing twins that are formed during HIPing interact and form either higher order twins or a \( \Sigma 1 \) boundary (i.e., form \( \Sigma 3^n \) boundaries; \( n \) can be 0 or >1; see Ref. [28]). As a result, the triple

Table 1

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cr</th>
<th>Mn</th>
<th>Mo</th>
<th>Ni</th>
<th>P</th>
<th>Si</th>
<th>C</th>
<th>S</th>
<th>N</th>
<th>O</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder</td>
<td>16.44</td>
<td>1.32</td>
<td>2.08</td>
<td>10.14</td>
<td>0.023</td>
<td>0.57</td>
<td>0.018</td>
<td>0.002</td>
<td>0.998</td>
<td>0.02</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Fig. 1. (a) Particle size distribution of the powder and (b) a typical HIPing cycle used in the present study. Temperature and pressure were ramped down after reaching 950 °C and 103 MPa, respectively.
Fig. 2. Grain boundary misorientation maps of as-received powder particles of different sizes (35 μm, 90 μm, and 225 μm) are shown in (a), (b), and (c). The grain boundaries are colour coded according to the legend shown in (e1). The legend is constructed using stereographic projection of surfaces of constant misorientation angle (θ) where each section is a standard stereographic triangle. The angle and axis information of any grain boundary can be obtained by matching its colour to the misorientation angle from the stereographic triangle and its position in the triangle, respectively. Only few sections are shown for illustration. Specific examples are shown in (e2), where the positions for P3, P9, and P27 boundary colours are marked using circles on 60°, 38.9°, 31.6°, and 35.4° misorientation surfaces. For a comparison, (c) is shown with only Σ3, Σ9, and Σ27 boundaries highlighted (with a tolerance angle of 3° from ideal misorientation) in red, yellow, and blue, respectively in (d). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
junctons with twin boundaries also evolve during HIPping. The evolution of number and length fractions of $\sum 3$, $\sum 9$, and $\sum 27$ and other special boundaries identified using the CSL theory framework ($\text{CSL} < 29$), and the distribution of triple junctions (i.e., $J_0$, $J_1$, $J_2$, and $J_3$) as a function of HIPping temperature are shown in Fig. 6. As seen in Fig. 6a, there is an increase, both in number and length fractions, in the CSL boundaries, with the increase in HIPping temperature. Fig. 6b shows distribution of triple junction types as a function of HIPping temperature. Triple junctions containing subgrain boundaries were not considered in the analysis. A decrease in the fraction of $J_0$ and increase in the fractions of other triple junctions is seen as the HIPping temperature is increased. The changes are more apparent for $J_0$, $J_1$, and $J_3$ fractions while the variation in $J_2$ with HIPping temperature is less pronounced. These observations are in accord with the increase in the fraction of $\sum 3^\circ$ boundaries as a function of HIPping temperature (Fig. 6a). In other words, as the fraction of twin boundaries (i.e., $\sum 3$, $\sum 9$, and $\sum 27$) increases, so will the fraction of triple junctions containing them.

During the early stages of HIPping, particles are deformed by the application of pressure at high temperature (i.e., they plastically yield), resulting in the formation of dislocations in the deformed particles. Since the deformation is at high temperature, the defect microstructure is simultaneously annealed. Formation of twin boundaries during high temperature deformation of the particles and a gradual increment in their fraction at progressively increasing temperatures (Figs. 4 and 6a) indicates the occurrence of dynamic recrystallization (DRX). In order to understand the progression of DRX, a criterion based on grain orientation spread (GOS)\(^1\) was applied on the EBSD data of the specimens at each HIPping condition. A value for GOS (measured in degrees) which can differentiate the recrystallized grains from the deformed grains was obtained from the EBSD data of three different heats of fully consolidated and solution annealed samples (i.e., which are fully recrystallized). Any threshold value between $1^\circ$ and $2^\circ$ gave similar results in all specimens, with more than $99\%$ of the area seen as recrystallized. However, sensitivity analysis on partially consolidated specimens revealed that the recrystallized fraction changed with the threshold value used. Specifically, with a change in the threshold value from $1.5^\circ$ to $2^\circ$, recrystallized fractions differed by about $20\%$ for the samples HIPed at $950 \, ^\circ\text{C}$ and $1000 \, ^\circ\text{C}$, while the same for $1050 \, ^\circ\text{C}$ and $1120 \, ^\circ\text{C}$ samples, it was less than $4\%$. However, the change in recrystallized fraction was much larger for a change in the threshold value from $1^\circ$ to $1.5^\circ$. So, a $1.5^\circ$ threshold for GOS was used for obtaining the DRX fractions in the partially HIPed specimens.

Fig. 7a shows the evolution of DRX and consolidated fractions obtained using EBSD data and image analysis, respectively, as a function of HIPping temperature. The consolidated fraction (or porosity fraction) from the optical images of the specimens at each HIPping condition was estimated using ImageJ software\[44]. Due to the contrast difference between pores and the bulk specimen in the optical images, thresholding to obtain binary images was straightforward. It is seen that the DRX fraction increases with the HIPping temperature. For the specimen HIPed at $1120 \, ^\circ\text{C}$, though little to no porosity was observed in the microstructure (in other words, the specimen was nearly consolidated), it did not undergo complete recrystallization (i.e., it was $\approx 93\%$ recrystallized). This suggests that the dwell time of 4 h employed during the HIPping cycle (which is part of a standard HIPping cycle) further promotes recrystallization. This result is in accord with the evolution of DRX fraction as a function of annealing twins, which is shown in Fig. 7b. From the state where the specimen is HIPed at $1120 \, ^\circ\text{C}$ (no dwell time) to fully consolidated condition, annealing twins are still formed. This can be seen from the increase in their number fraction, i.e., from $23\%$ to $26\%$ (Fig. 6b). Also, the evolution of DRX fraction follows the number fraction of annealing twins at different HIPping temperatures (i.e., DRX fraction increases with the increase in the twin fraction), which suggests that during HIPping, this material recrystallizes by twinning.

TRDs, which are linked to recrystallization, were reconstructed for the fully consolidated specimen, and are shown in Fig. 8a with $\sum 3^\circ$ boundaries depicted as per the legend shown in Fig. 8b. A visual inspection of the map reveals the sizes of TRDs which, following the previous proposition\[23,33\], can be treated as the classical grain size, and the random boundaries that highlight the paths along which cracks propagate. For the partially HIPed specimens, along with the TRD reconstruction, additional TRD parameters were also obtained; these are shown in Table 3. The average values of all TRD parameters increase as the HIPping process progresses. Specifically, for the specimen HIPed at $950 \, ^\circ\text{C}$, ($N_2$) is close to 1, with ($\text{LLC}$) $= 0.08$; this means that most of the grains have not yet twinned (in other words, they have not recrystallized). This result can be correlated with low fractions of twin boundaries (see Fig. 6a). In the fully consolidated specimen, which has completely recrystallized, average values of TRD parameters have increased compared to the sample HIPed at $950 \, ^\circ\text{C}$. Interestingly, albeit the sample is nearly consolidated by $1120 \, ^\circ\text{C}$ (without dwell time at that temperature), ($N_2$) and ($\text{LLC}$) values have increased after HIPping at $1160 \, ^\circ\text{C}$ (i.e., full HIP cycle); additionally, $\text{LLC}_{\text{max}}$ has increased from 7 to 9 after the full HIPping cycle. This clearly shows that the sample recrystallizes during the 4 h dwell time (in other words, twin chains in TRDs have propagated further), and is in agreement with the results shown in in Fig. 7. Increase in the (TRD) size from 14.2 $\mu$m to 35 $\mu$m indicates grain growth during the dwell time of HIPping cycle.

The frequency distributions for TRD size, $N_2$, and LLC for each HIPping condition, which effectively reflect their evolution during the HIPping process, are shown in Fig. 9. The TRD size distribution curves are seen to shift to the right as the function of HIPping temperature (Fig. 9a), resulting in an increase in the (TRD) size. A

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\[^1\] GOS is defined as “the average difference in orientation between the average grain orientation and all measurements within a single grain”[43].
comparison between the average particle size of as-received powder and the average TRD size in fully consolidated specimen (80 μm and 35 μm, respectively) suggests that the length scale of TRDs will be less than the particle size. The distribution of LLC is shown in Fig. 9b. Not only does the LLC_max increase (also see Table 3), but the number of TRDs with LLC > 0 also increases. Specifically, in the specimen HIPed at 950 °C, less than 1% of TRDs have LLC ≥ 2 (LLC_max = 5), while for the completely consolidated specimen, 33% TRDs have LLC ≥ 2 (LLC_max = 9). Similar observations can be made for the distribution of N_g, shown in Fig. 9c.

4. Discussion
4.1. Microstructural evolution during HIPing
4.1.1. Evolution of CSL boundaries and triple junctions
Grain boundary misorientation maps (Fig. 2) and the frequency of CSL boundaries (3 ≤ Σ ≤ 29) in the powder shown in Table 2, which was averaged over 3 particles of different sizes, indicate that the microstructure is dominated by high angle grain boundaries. Most of the Σ 3 boundaries in the powder particles had deviations

### Table 2
CSL boundary fractions and triple junction distributions averaged from the data of the three powder particles shown in Fig. 3, and for the fully consolidated specimen. Values in the brackets indicate standard deviation.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Σ 3</th>
<th>Σ 9</th>
<th>Σ 27</th>
<th>Total CSL (Σ ≤ 29)</th>
<th>J0</th>
<th>J1</th>
<th>J2</th>
<th>J3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder</td>
<td>L^a</td>
<td>N^b</td>
<td>L</td>
<td>N</td>
<td>L^c</td>
<td>N</td>
<td>L</td>
<td>N</td>
</tr>
<tr>
<td>FC</td>
<td>53 (± 1)</td>
<td>36 (± 1.2)</td>
<td>1.56 (± 0.1)</td>
<td>3 (± 0.15)</td>
<td>1 (± 0.1)</td>
<td>60 (± 1.5)</td>
<td>32 (± 1.4)</td>
<td>25 (± 2)</td>
</tr>
</tbody>
</table>

^a Length fraction.  
^b Number fraction.  
^c Fully consolidated.

![Fig. 4. Grain boundary misorientation maps of 316L HIPed at a pressure of 103 MPa and at increasing temperatures, starting at 950 °C. The grain boundaries are colour coded according to the legend shown in Fig. 2e. The fraction of subgrain boundaries, shown in white colour, gradually decreases with concomitant increase in the fraction of annealing twins as the HIPing temperature increases. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)](image-url)
fractions of twin boundaries (i.e., $f_3^3$ and $f_3^9$, respectively, of $\Sigma 3$, $\Sigma 9$, and $\Sigma 27$) in the fully consolidated specimen, it is seen that $f_3^3 < f_3^9$, $f_3^9 > f_3^19$, and $f_3^{27} > f_3^{127}$. Since the majority of CSL boundaries consists of $\Sigma 3$, $f_3^3 < f_3^9$ (see Table 2). Such differences between length and number fractions in CSL boundaries have previously been reported in the literature [20–22,45].

Since the $\Sigma 3$ boundaries are straight and long (i.e., annealing twins), they are, on average, longer than other high angle grain boundaries, thus giving rise to the observed inequality (i.e., for $\Sigma 3$ and total CSL fraction) [21]. It has been suggested that the constraint imposed by the crystallography of the triple junctions necessitates the presence of $\Sigma 9$ and $\Sigma 27$ boundaries in the microstructure and that they have no energetic preference among other non-$\Sigma 3$ CSL boundaries for their nucleation [46] (relatively very few $\Sigma 9$ and $\Sigma 27$ boundaries were observed at $J_3$ in this study). Since the length per boundary of such crystallographically necessary boundaries at the triple junctions containing $\Sigma 3$ boundaries is often very small, it translates to $f_3^9$ being greater than $f_3^3$ for $\Sigma 9$ and $\Sigma 27$ boundaries.

The distribution of triple junctions has also evolved accordingly (Fig. 6b). Specifically, a decrease in the fraction of $J_0$ and an increase in the fraction of $J_1$, $J_2$, and $J_3$ junctions is seen, which correlates well with the increase in the number fraction of twin boundaries. Experimental results on the microstructural characterization of several low to medium SFE energy FCC materials have clearly demonstrated the non-random nature of the distribution of triple junctions, and have shown that it is related to the crystallographic constraints imposed at the triple junction [22,24,25,47].
The average size of TRDs was 35 μm. The boundaries are coloured according to the legend shown in (b), where the numbers indicate $n$ in $\sum^3$. The average number of grains in the TRDs was 3.77 while the average size of TRDs was 35 μm. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

### 4.1.2. Dynamic recrystallization and the development of TRDs during HIPing

In materials with low to medium SFE, elevated temperature deformation results in DRX [49,50]. In these materials, it has been shown that the formation of high-order twin chains in single and polycrystals is a key feature of the recrystallized microstructure, and that twinning is an active nucleation mechanism for recrystallization [23,51–55]. Although the present investigation was not aimed towards providing any additional insights on DRX, already established mechanism, i.e., twinning during DRX, is observed during HIPing of 316L powder.

The fraction recrystallized as a function of HIPing temperature was estimated using GOS criterion (Fig. 7a). It must however be noted that this fraction, which is around 38% for the specimen HIPed at 950 °C, also includes regions that have not undergone DRX. Specifically, regions within large particles, or particles themselves, that have features of the as-received state (in other words, they have not undergone deformation), yet having GOS < 1.5° were observed. While in principle these regions have not recrystallized, they were treated to be part of the DRX region since their GOS value is < 1.5°. This overestimation in DRX fraction decreases with increasing HIPing temperatures as most of the particles would have already deformed, and hence would have either recrystallized, or be in the deformed state. This, however, is dependent on the particle size distribution and applied pressure, as will be discussed in the next section.

In regards to recrystallization during HIPing, an important observation can be made from Fig. 7 and Table 3. The pressure used for HIPing the powder at different temperatures (i.e., 103 MPa) is high enough for them to plastically yield. Plastic deformation of the particles is only possible during early stages of HIPing (i.e., at 950 °C and 1000 °C in this study), where contact stresses between the particles are high, and is the main factor contributing to the densification. Therefore, the stored energy due to the plastic deformation of particles at early stages contributes to recrystallization at higher temperatures, which is because, there is little porosity at higher temperatures for the compact to deform as most of the densification has already happened. It is then the case of static recrystallization (SRX) and/or strain induced boundary migration (SIBM) contributing to microstructural changes during final stages of HIPing (i.e., after 1050 °C and during dwell time). In other words, during HIPing, the microstructure evolves via dynamic and static recrystallization. However, distinction has not been made in the present study.
It is observed that the propagation of twin chains during HIPing, as seen from the evolution of $\langle \text{LLC} \rangle$ and $\text{LLC}_{\text{max}}$, makes the specimen more polysynthetic (refer Table 3). In other words, during HIPing process, as the specimen recrystallizes, reverse twinning is promoted. Lind et al. [27] analyzed TRDs in 3D using near-field high-energy diffraction microscopy (nf-HEDM) on a synchrotron source in a normal and a grain boundary engineered copper sample, respectively, and demonstrated that grain boundary engineered sample is more polysynthetic than the normal sample. Liu et al. [56], in a grain boundary engineered nickel based alloy, have demonstrated that multiple twinning results in the formation of back and forth pattern (in other words, both higher and lower generations of twin orientations are produced). However, a strong preference for reverse twinning (i.e., polysynthetism) and hence, certain orientations was observed. It thus appears that multiple twinning, regardless of the processing condition, results in the material becoming more polysynthetic.

4.2. Factors influencing the development of grain boundary network in HIPed 316L steels

Size dependent inhomogeneous nature of plastic deformation of particles is an important aspect during HIPing, which affects the final microstructure. Specifically, Fig. 5 clearly demonstrates that smaller particles deform more than larger particles. This result is in accord with the ones reported in other investigations [57–60], and can be rationalized based on the fact that the fraction of contact area to the available surface area is higher for smaller particles than for larger particles. As illustrated by Wright et al. in their HIP model, small particles will see increased deformation if present in interstices of an arrangement of large particles [61]. In addition, the mechanical properties of the powder particles potentially vary depending on their size. Specifically, if we consider two different powder sizes shown in Fig. 2 (i.e., 35 μm and 225 μm) and their average grain size, the larger particle contains an order of magnitude more number of grains than the smaller particle. Consequently, it is likely that larger particles would be harder than smaller particles because they contain many more grains that constrain each other during deformation. So, even with the theoretical density achieved after a full HIPing cycle, depending on the particle size distribution, some non-deformed particles can still remain in the compact. In other words, they would just retain their original shape, and won’t undergo recrystallization; this is illustrated in Fig. 11a. It shows the reconstructed TRDs for a region in a fully consolidated specimen that has not completely recrystallized (i.e., a powder particle is partially in its original state). The region surrounding the particle has recrystallized, as evidenced by the presence of annealing twins. In
order to see if the as-received powder when annealed at high temperature undergoes recrystallization, it was put in a capillary and heat treated at \(1100\,^\circ\text{C}\) for 15 min under argon atmosphere. Comparing the grain boundary network of the heat treated powder (shown in Fig. 11b) with that of non-deformed region in the fully consolidated specimen (Fig. 11a), it is seen that they are very similar. This further suggests that the as-received powder does not have enough stored energy for it to recrystallize if it has not deformed, albeit subjected to full HIPing cycle.

So, it can be understood that a temperature cycle without simultaneous (or prior) deformation of the particles would only result in grain boundary migration and perhaps grain growth, and that deformation of the particles is a prerequisite for recrystallization (compare Fig. 11b with the as-received powder shown in Fig. 2). It must be noted that this is not a universal feature of gas-atomized powders; it has recently been demonstrated that powders of titanium aluminide undergo recrystallization even with a simple heat treatment without prior plastic deformation \[62\]. Specifically, Guyon et al. have shown that the elastic coherency strain and interfacial energy in the particles provide the driving force for recrystallization even in the absence of prior plastic deformation \[62\].

While the tendency of a material to twin depends primarily on the chemistry (in other words, SFE), thermomechanical processing has a second order, but strong, effect (e.g. \[45\], and references therein). Similar to the case where the grain boundary networks in low to medium SFE materials produced from solidification route strongly depend on their thermomechanical processing history, grain boundary network of HIPed 316L depends strongly on particle characteristics and processing parameters. Liu et al. \[34\] have studied the effect of initial grain size on the development of grain boundary network during grain boundary engineering (GBE) of alloy 690. Besides showing the effect of pre-strain level on the recrystallized microstructure, it was also demonstrated that a large initial grain size increases the TRD size but reduces the twin boundary density, and a small initial grain size induces higher twin boundary density, but higher random boundary density and smaller TRD size. This in principle applies to HIPed 316L. Here, the size distribution of powder particles, their grain size, the extent they are strained to, the temperature, and time, affect TRD development.

Specifically, the average grain size of the powder depends on the size of the powder; large particles have relatively larger grain size than the small particles (Fig. 2). The level of strain experienced by the particles depends on their packing fraction (in other words, their tap density), which in turn is governed by the particle characteristics (size distribution and morphology) and the applied pressure. Specifically, high packing fractions result in low shrinkage of the compact and therefore low strains, while low packing fractions result in high shrinkage and high strains. The importance of dwell time was highlighted previously. Specifically, it was observed that twin chains in the TRDs propagate further during the 4 h dwell time of the HIPing cycle (see Table 3). With particle characteristics unaltered, the effect of decreasing or increasing the dwell time on final microstructure needs further investigation.

4.3. Possibilities of grain boundary control in NNS PM-HIPed components

Thermomechanical processing of cast materials allows the realization of a variety of microstructures and hence, a range of
properties. For low to medium SFE materials (e.g., 316L, 304L, alloy 690), control of grain boundary network using various strain-anneal or strain-recrystallization processes that result in $\sum 3$ and high-order twin boundaries to be part of the grain boundary network has been shown to improve their performance. However, in the case of powder-HIP manufacturing, only post-HIP heat treatments are possible if the principal objective is to achieve near net shape. Preceding discussion on how the grain boundary network evolves during HIPing, and the factors affecting it, offers some potential directions that could be pursued to exercise control over the development of grain boundary network. Since these changes can be applied during the HIPing process, they can be implemented on near net shape components. Two examples are presented. It must be noted that the aim here is to only demonstrate that the topology of the grain boundary network can be changed by altering the traditionally used HIPing cycle; it is an optimization problem and no attempts were made towards the same in the present study.

Fig. 12 shows the grain boundary misorientation map of the sample HIPed at 950°C (without any dwell time), which was subsequently annealed for 10 min at 1100°C (i.e., post-HIP). A comparison with the microstructure of 950°C as-HIPed specimen (see Fig. 4a) reveals that the heat treated specimen has undergone

Table 4
A comparison of triple junction distributions and the average TRD parameters for four specimens, highlighting the effect of heat treatments.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$J_0$</th>
<th>$J_1$</th>
<th>$J_2$</th>
<th>$J_3$</th>
<th>$\langle TRD \rangle$</th>
<th>$\langle N_p \rangle$</th>
<th>$\langle LLC \rangle$</th>
<th>$LLC_{max}$</th>
<th>$\langle p \rangle$</th>
<th>$p_{max}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>950HIP</td>
<td>78</td>
<td>17</td>
<td>2</td>
<td>3</td>
<td>4.49</td>
<td>1.08</td>
<td>0.08</td>
<td>5</td>
<td>1.0</td>
<td>2.0</td>
</tr>
<tr>
<td>950HIP + 10mHT</td>
<td>38</td>
<td>46</td>
<td>5</td>
<td>11</td>
<td>15</td>
<td>2.51</td>
<td>1</td>
<td>8</td>
<td>1.13</td>
<td>2.75</td>
</tr>
<tr>
<td>FC</td>
<td>15</td>
<td>59</td>
<td>8</td>
<td>18</td>
<td>31</td>
<td>4.2</td>
<td>1.57</td>
<td>8</td>
<td>1.3</td>
<td>3.33</td>
</tr>
<tr>
<td>FC + 66hrHT</td>
<td>1</td>
<td>45</td>
<td>4</td>
<td>50</td>
<td>156</td>
<td>16.81</td>
<td>2.13</td>
<td>9</td>
<td>1.57</td>
<td>6.12</td>
</tr>
</tbody>
</table>

Fig. 13. (a) Grain boundary misorientation map of a fully consolidated specimen with 95 ppm of oxygen. Parent grains of TRDs are shown in (b). Grain boundary misorientation map and parent grains of TRDs of the same specimen after annealing at 1100°C for 66 h are shown in (c) and (d), respectively. Changes in the grain boundary network are apparent. The grain boundaries in (a) and (c) are colour coded according to the legend shown in Fig. 2e. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
The aim of the present study was to understand the evolution of grain boundary network in 316L austenitic steel during HIPing. The main findings are:

- The as-received nitrogen gas atomized powder predominantly contained a network of random boundaries while the completely consolidated HIPed material had a large fraction of annealing twins, indicating that the principal mechanism governing the microstructural evolution during HIPing is recrystallization (DRX and SRX).
- As-received powder does not have enough stored energy to recrystallize without deformation. Plastic deformation of the particles, which occurs at high temperature during early stages of HIPing, is a prerequisite for recrystallization. Because of the size dependence on the extent of their deformation, particle size distribution strongly influences the final microstructure.
- The recrystallized fraction increases during both ramping up stage (i.e., of P and T) as well as during the dwell time of the HIPing cycle, and correlates well with the evolution of number fraction of $\sum 3$ boundaries. While the fraction of triple junctions containing $\sum 3$ boundaries increases concomitantly, they are predominantly part of $J_3$ triple junctions.
- Quantitative analysis of TRDs, which are linked to recrystallization, reveals that $\langle T_{d} \rangle$, $\langle N_{d} \rangle$, $\langle LLC \rangle$, and $\langle \rho \rangle$ increase during HIPing.
- By altering the particle characteristics, HIPing cycle, and post-HIP heat treatments, it is possible to change the grain boundary network, indicating the potential for grain boundary engineering during HIPing.

Finally, it must be recognized that HIPing is a thermomechanical process. While in most cases, the primary objective of powder based HIPing is to produce a fully dense component, of significant importance is the microstructural evolution during HIPing and the topology of the grain boundary network in the fully consolidated material. Powder characteristics (particle size distribution, grain size, morphology, tap density, chemistry, etc.), the HIPing cycle, and post-HIP heat treatment have a critical role to play in the development of the final microstructure. For materials which profusely twin (e.g., 316L, alloy 600 and 690), even with the constraints imposed by the way in which pressure and temperature can be applied, HIPing process can potentially be tailored to produce increased fractions of twin boundaries that are part of the grain boundary network. Such an optimized process is of great value because of the added benefit of the component being of near net shape.

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References
