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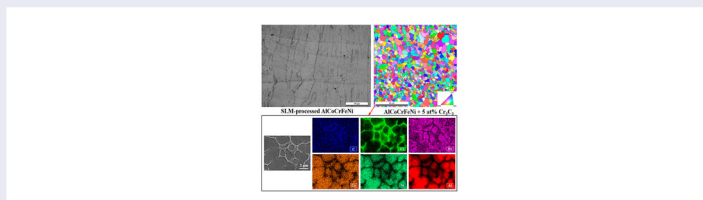
Promoting columnar-to-equiaxed transition in AlCoCrFeNi high entropy alloy during selective laser melting by adding Cr₃C₂

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ABSTRACT

The 5 at% Cr₃C₂ particles were mixed with AlCoCrFeNi high entropy alloy powder particles and then selectively laser melted. The addition caused full columnar-to-equiaxed grain transition (CET) and significant grain refinement. The inter-dendritic regions and grain boundaries (GBs) of the as-fabricated sample were segregated with Cr and C, which led to the formation of an FCC belt/network structure along GBs. The grain interior was dominated by BCC/B2 and sporadically decorated by Cr₇C₃ particles. The partitioning of Cr and C into liquid ahead of the solidification front, which may have generated significant constitutional supercooling, is believed to be responsible for the full CET.



IMPACT STATEMENT

The present study introduces a small fraction of Cr₃C₂ particles into AlCoCrFeNi high entropy alloy and successfully enables full columnar-to-equiaxed grain transition and significant grain refinement during selective laser melting.

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KEYWORDS

Selective laser melting; high entropy alloy; columnar-to-equiaxed transition; grain refinement; segregation

1. Introduction

Selective laser melting (SLM) is well known for its outstanding near-net-shape manufacturing capability that offers great design flexibility and enables a high level of product customization. On the other hand, it is also known for its unique processing characteristics such as high thermal gradients in melt pools, high cooling rates and complex thermal cycling [1–3]. The steep thermal gradients within melt pools usually result in epitaxial grain growth and thus in the formation of coarse columnar grains and strong texture that can cause mechanical property anisotropy [4–9]. The inhomogeneous thermal distribution and high cooling rates within a build during SLM usually cause the development of high internal stresses, which, together with a large columnar grain structure, can lead to hot tearing in some kinds of metallic materials [7–15]. It is very common in a number of high-strength nickel-based superalloys [7,8], aluminum

alloys [9–11], and titanium aluminides [12,13]. It has also been observed in SLM-processed high-strength high entropy alloys (HEAs), such as AlCoCrFeNi [14–18], which are usually considered promising for structural or engineering applications [14–18]. Owing to the hot tearing issue, the near-net-shape manufacturing potential of SLM cannot be fully exploited to expand the application of high-strength HEAs.

One effective strategy to deal with hot tearing is to promote columnar-to-equiaxed transition (CET) and grain refinement during SLM. So far, the most common approach to promote CET during SLM is to introduce potent nucleants to promote the formation of new equiaxed crystals. This route has been proven to be particularly effective for SLM-processed aluminum and its alloys. Al₃X-forming elements, such as Zr, Sc, Ti, Ni, and Nb, were usually added to aluminum or its alloys prior to SLM processing. Potent heterogeneous nucleants, Al₃X

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(i.e. X = Zr, Sc, Ti, and Ni), which constitute low mismatch with the matrix, were then formed during SLM and significantly promoted the CET and grain refinement [9,19–22]. Concurrent with this effect is the full suppression of hot tearing during SLM of aluminum alloys. Many other metallic materials, such as nickel-base superalloys and HEAs, however, are less lucky with this approach due to the lack of suitable and potent inoculants.

Recently, the addition of carbon into high or medium entropy alloys, such as AlCoCrFeNi, CrFeCoNi, and CrCoNi, through the conventional melting and casting method was found to have promoted grain refinement by enhancing constitutional supercooling in the liquid ahead of the solidification fronts [23–25]. For example, the addition of C in AlCoCrFeNi through casting led to the segregation and accumulation of C along grain boundaries during solidification, which is not only good for the development of large constitutional supercooling and thus grain nucleation but also helpful in inhibiting grain growth [24]. Li et al. [26] found that the addition of WC particles into CoCrFeMnNi HEA through direct laser deposition (DLD) led to remarkable grain refinement by forming carbide particles during solidification that may have acted as effective heterogeneous nucleation sites. However, it is noted that there is a lack of reports on the influence of the addition of C or carbides on the microstructural evolution of HEAs during SLM. It is well established that as compared with conventional casting and DLD, SLM usually demonstrates much higher cooling rates (10^4 – 10^6 °C/s) and higher thermal gradients within melt pools. With such extreme processing characteristics, whether the addition of carbon or carbides can either enhance constitutional supercooling ahead of the solid/liquid front or lead to the formation of potent carbide nucleants during solidification so that both CET and grain refinement of HEAs can be achieved remains unknown. So does the effectiveness of the addition in improving the SLM processability of AlCoCrFeNi. Thus, in this study, we add a small amount of Cr₃C₂ into AlCoCrFeNi through powder mixing and SLM to investigate the role of the addition in the microstructural evolution and solidification behavior of the alloy. The influence of the addition on the SLM processability of the alloy and mechanical properties will also be examined.

2. Materials and methods

Argon atomized equimolar AlCoCrFeNi HEA powder with a particle size range of 15–53 µm and Cr₃C₂ powder a particle size range of 2–3 µm were used in the present study. According to a previous study [24], the addition of 2 at% C into AlCoCrFeNi HEA through the conventional casting method can result in significant

grain refinement and improvement in yield strength and compressive strength of the HEA. Thus, in this study, 5 at% Cr₃C₂ powder particles, equal to addition of 2 at% C, were mixed with the HEA powder particles (referred to as HEA-Cr₃C₂ hereinafter) in an argon atmosphere using a horizontal ball mill (Figure 1(a)) [27]. A ball-to-powder weight ratio of 4:1 and balls with a diameter of 5 mm were used for the powder mixing. The milling process took 20 min with a shaft rotation speed of 600 rpm. Both the pure HEA powder and the HEA-Cr₃C₂ powder mixture were then processed using a Renishaw AM 400 system. Samples with a dimension of 10 × 10 × 5 mm³ were built under a modulated pulsed laser mode in an argon atmosphere for microstructural characterization. A laser power of 400 W, a point exposure duration of 90 µs, and a powder layer thickness of 50 µm, which led to a very low porosity level (< 0.1%), were selected to process the powder materials. The point distance and hatch distance were set to be 60 and 90 µm, respectively. The as-fabricated samples were longitudinally sectioned by electrical discharge machining (EDM) and then ground using SiC papers from 320 grit up to 4000 grit before being polished using 3 µm diamond suspension and colloidal silica suspension (or OPS solution). Some of the as-polished samples were analyzed using the electron backscattered diffraction technique (EBSD) with a step size of 500 nm. The samples were then etched and examined using optical microscopy (OM) and scanning electron microscopy (SEM). A Leica DM4000 OM machine and a Zeiss SUPRA55 SEM machine, and an FEI Quanta 450 SEM machine, which was equipped with an EBSD detector, were used for the microstructural characterization. Transmission electron microscopy (TEM), high-resolution TEM (HRTEM), and energy dispersive X-ray spectroscopy (EDS) studies were also performed on some of the as-fabricated samples. Disc specimens of 3 mm in diameter were machined out of the samples and ground into a thickness of 50–100 µm and then electro-polished or ion beam milled to perforation using a Gatan 691-type ion beam thinner. The TEM and HRTEM studies were conducted using an FEI TecnaiG20 FEG TEM microscope fitted with an Oxford EDS detector. The Vickers hardness tests were conducted in a Future-Tech FM-800 microhardness testing system using a load of 300 gf and a dwell time of 20 s.

3. Results and discussion

Figure 1(b,c) shows the particle distribution and morphologies of the as-received HEA powder and the HEA-Cr₃C₂ powder mixture, respectively. It is obvious that the as-received HEA powder shows a homogeneous particle size distribution and these particles are generally

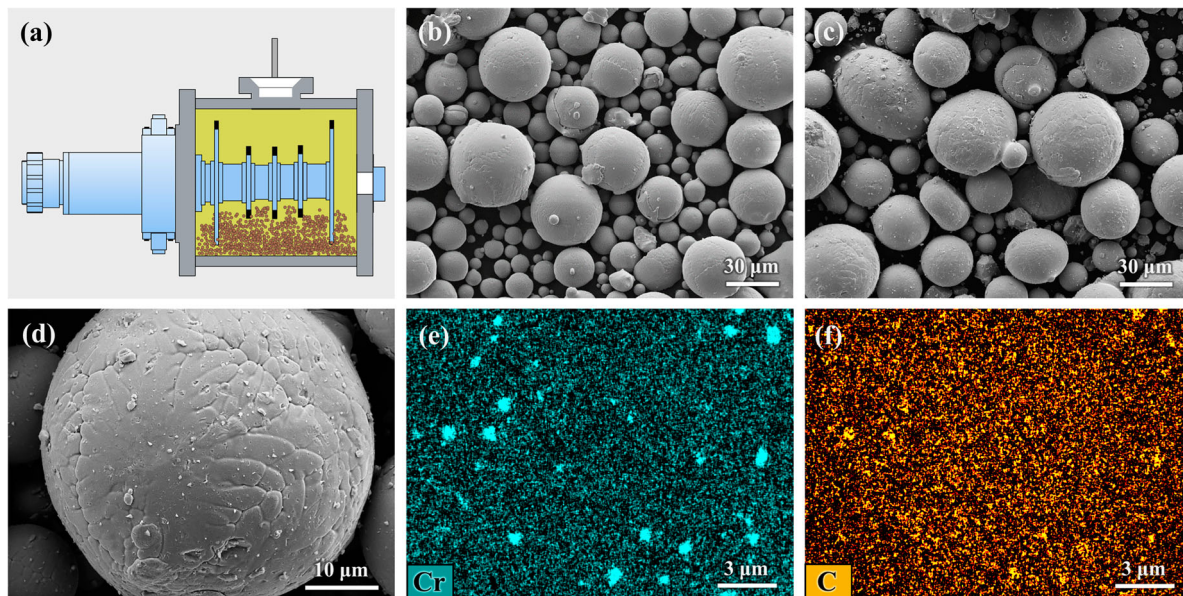


Figure 1. (a) Schematic illustration of the horizontal ball mill used in the current study; SEM micrographs showing particle distribution and morphology of (b) the as-received HEA powder and (c) the HEA- Cr_3C_2 powder mixture, (d) particle surface structure of the HEA- Cr_3C_2 powder mixture; (e and f) EDS mapping result of the surface of a HEA particle assembled by fine Cr_3C_2 powder particles shown in (d).

spherical or near-spherical, which are good for powder spreading during SLM. After short-duration mixing with the 5 at% fine Cr_3C_2 particles using the horizontal ball mill, the majority of the HEA particles remain spherical or near-spherical as shown in Figure 1(c,d). The ultrafine Cr_3C_2 particles have been uniformly assembled onto the HEA powder particle surfaces, as shown in Figure 1(d–f), which would be undoubtedly beneficial for the development of good chemical and microstructural homogeneity during the subsequent SLM processing. In addition to EDS mapping analysis, some of the ultrafine particles adhered to the large powder particle surfaces were also subjected to point EDS analysis and the results are included in Table S1. It is shown that the ultrafine particles are mainly enriched in Cr and C, further confirming that they are indeed Cr_3C_2 particles. In contrast, the large particles mainly contain Al, Co, Cr, Fe, and Ni elements and show a near equiatomic proportion, indicating that they are the AlCoCrFeNi HEA powder particles.

Figure 2 shows the grain structures along the longitudinal sections of the as-built HEA and HEA- Cr_3C_2 samples. It is obvious that the HEA sample is dominated by large columnar grains, as shown in Figure 2(a,b). This is the typical microstructure of SLM-processed AlCoCrFeNi, which is the result of steep thermal gradients and rapid cooling rates of melt pools during solidification and is consistent with previous reports [16,28]. Meanwhile, with the formation of large columnar grains and potentially high internal stress, many cracks were observed to be along many columnar grain boundaries (Figure S1(a,b)). With the addition of Cr_3C_2 , the large

columnar grains disappeared, and ultra-fine equiaxed grains formed (Figure 2(c,d)). The sizes of the equiaxed grains in the HEA- Cr_3C_2 sample are generally several microns, which are two orders of magnitude smaller than those in the Cr_3C_2 -free HEA sample. The results indicate that both CET and grain refinement have been completely activated during SLM with the addition of Cr_3C_2 . With the significant grain refinement, the grain boundary cracks associated with large columnar grains have been completely suppressed. However, due to stress buildup at the root of the build during SLM, cracks still formed from the bottom and propagated upwards (Figure S1(c)). The upper regions are completely free from cracks (Figure S1(d)), thanks to the grain refinement. In the intact areas, the interlayer boundary regions tend to contain relatively larger equiaxed and polygonal grains as compared with the interior of solidified melt pools, which is generally dominated by finer dendritic grains (Figure 2(c,d)). This can be associated with thermal cycling and reheating on the previous solidified layers. The surface and subsurface regions of the previous solidified layer may have experienced relatively high-temperature remelting or reheating during the subsequent powder layer processing, which promoted coarsening and coalescing of fine grains in the interlayer interfacial regions.

Figure 3 shows the microstructural details in the HEA- Cr_3C_2 sample. Again, it is shown that the central region of a solidified melt pool is dominated by a fine dendrite structure (Figure 3(c,e)), while the interlayer boundary region mainly consists of relatively larger near-spherical or polygonal equiaxed

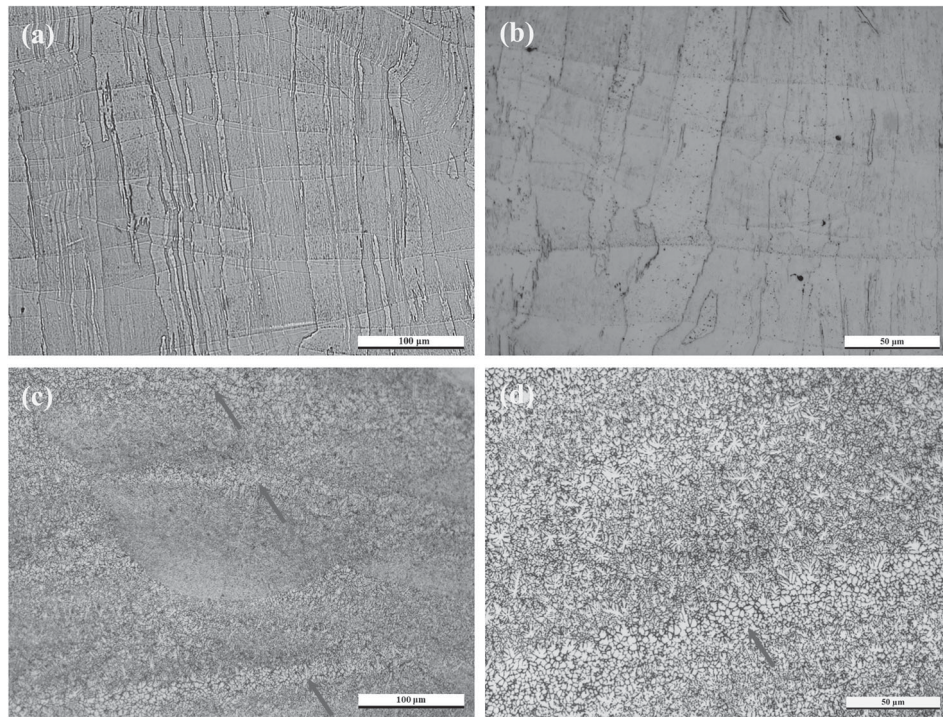


Figure 2. OM micrographs showing grain structures in the SLM-processed (a and b) HEA and (c and d) HEA-Cr₃C₂ samples. The arrows in (c) and (d) refer to interlayer boundary regions where relatively larger equiaxed grains dominate, in contrast to the finer grains present within the interior of solidified melt pools.

grains (Figure 3(d)). Both the grain boundary (GB) regions and inter-dendritic regions were found to contain belt or stripe structures (Figure 3(c–e)), leading to the development of a network structure along the GBs. According to EDS mapping results, as shown in Figure 3(f), these GB or inter-dendritic belt structures tend to be segregated with Cr and C. Point TEM-EDS analysis results in Table 1 further confirm that the belt structures are enriched in Cr and C but depleted in Al. The segregation of Cr and C along inter-dendritic regions and GBs indicate that these elements have been expelled into the liquid ahead of the solidification front during solidification. This is in agreement with the HEA-C phase diagram, which shows that the solute of C has a partition coefficient of $k < 1$ [23], which means that C would be rejected ahead of the solid–liquid interface during solidification. It is analogous to the segregation of Hf, Ti, Ta, and W at cell walls in SLM-processed CM247LC and the segregation of Cr and Mo at cell walls and GBs in SLM-processed 316L stainless steel as reported in Refs. [29–32]. The partitioning of Cr and C to the liquid, in turn, would generate constitutional supercooling in the liquid ahead of the solidification front and thus promote the nucleation of new equiaxed crystals. This should be the main reason for the acquisition of full CET in the present study. The enrichment of solutes in a diffusion layer ahead of the advancing solid/liquid interface is also

Table 1. Point TEM-EDS analysis results on a GB belt structure, a grain interior/matrix area, and a carbide particle in the SLM-processed HEA-Cr₃C₂ sample (in at%).

Element (at%)	Al	Co	Cr	Fe	Ni	C
Matrix area	15.92	19.09	21.85	20.87	21.14	1.14
GB belt structure	4.01	11.36	46.95	15.55	6.08	16.05
The particle shown in Figure 5(c)	0.07	1.31	27.10	2.53	0.13	68.87

believed to restrict grain growth since the solutes diffuse slowly [23]. The mechanism of achieving full CET and grain refinement in the present study is obviously different from the nucleant-induced heterogeneous nucleation mechanism that has been widely observed and applied in many previous studies, especially those on SLM-processed aluminum alloys [19–22]. As such, the current study provides a new strategy to promote CET and grain refinement during metallic SLM, i.e. promoting micro-segregation by adding particles or elements that are liable to partition to the liquid during solidification.

Figure 4(a) shows the EBSD inverse pole figure (IPF) color map of the grain structure in the HEA-Cr₃C₂ sample. It is clear that the sample consists of both fine equiaxed grain regions and relatively larger grain regions, which is consistent with the above OM and SEM observations. The grains tend to be highly randomly oriented and thus the texture tends to be really weak. This is also

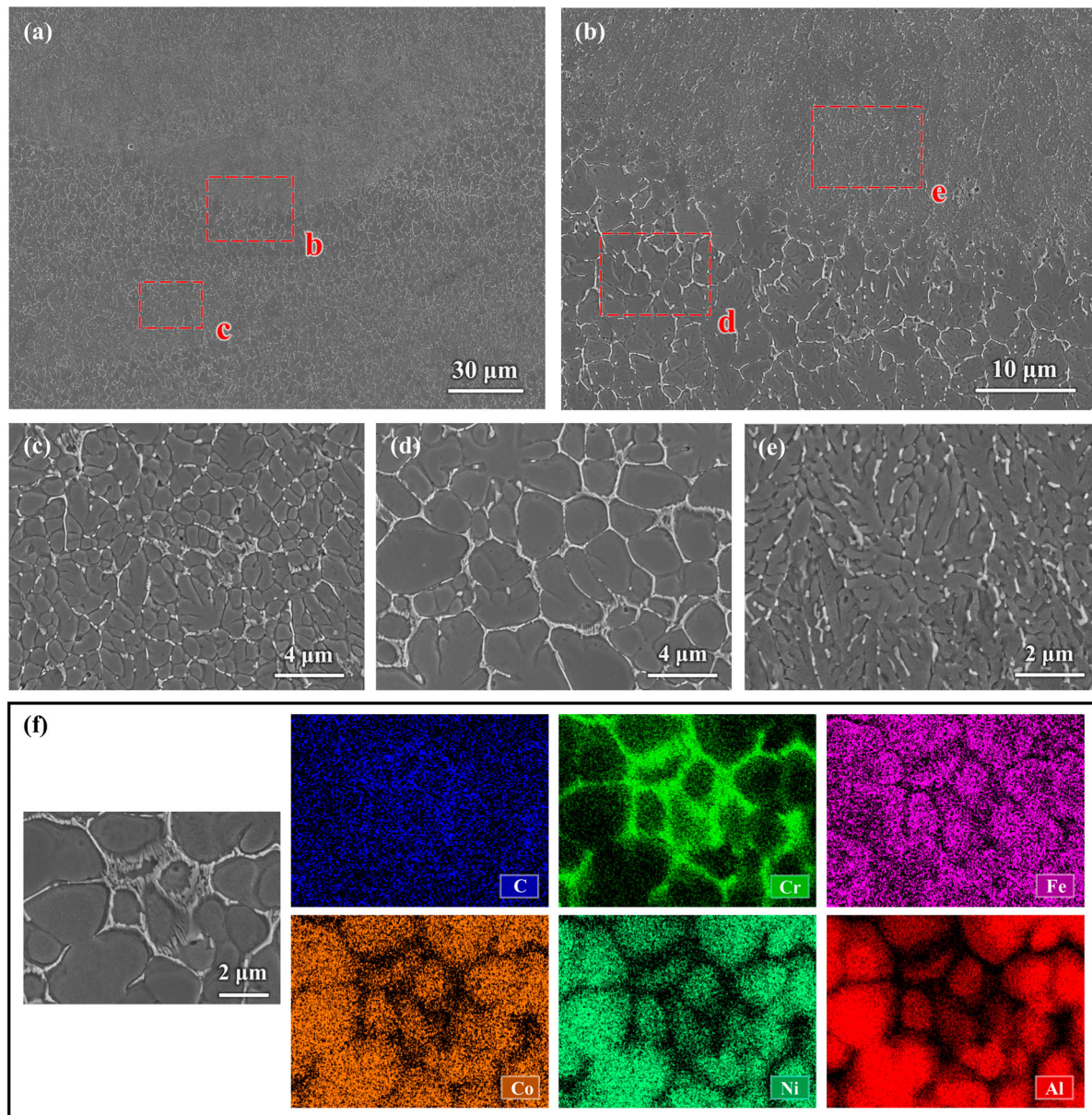


Figure 3. (a–e) SEM micrographs showing the microstructure in the SLM-processed HEA- Cr_3C_2 sample; (f) SEM micrograph and EDS mapping results on an interlayer boundary region of the SLM-processed HEA- Cr_3C_2 sample. Regions b and c in (a) correspond to the magnified micrographs of (b) an interlayer boundary region and (c) a region within a solidified melt pool, respectively. Regions d and e correspond to the magnified micrographs of (d) and (e).

confirmed by the very low maximum multiples of the uniform distribution (MUD) values in the pole figure shown in Figure 4(b). Quantitative analysis based on the EBSD data shows that the grain sizes in the HEA- Cr_3C_2 sample are between $0.5\ \mu\text{m}$ and $10\ \mu\text{m}$ in diameter with the majority of them falling between $2\ \mu\text{m}$ and $5\ \mu\text{m}$; see Figure 4(c). Moreover, there is a significantly high fraction of high angle grain boundaries (HAGBs) in the sample. According to Figure 4(d), more than 95% GBs are HAGBs (grain boundary angles $\geq 15^\circ$), which obviously accounts for the low texture level in the current sample,

thanks to the development of equiaxed grain structure. Quantitative analysis of the grain aspect ratio and grain shape circularity was also conducted; Figure 4(e,f). It is shown that grains with aspect ratio values larger than 0.3, which can be regarded as equiaxed grains, account for $> 96.4\%$ of all grains. According to Hunt [33], when the volume fraction of equiaxed crystals exceeds a critical value of 49%, the columnar growth will be prohibited and a fully equiaxed grain microstructure will be achieved. This is definitely the case for the current study.

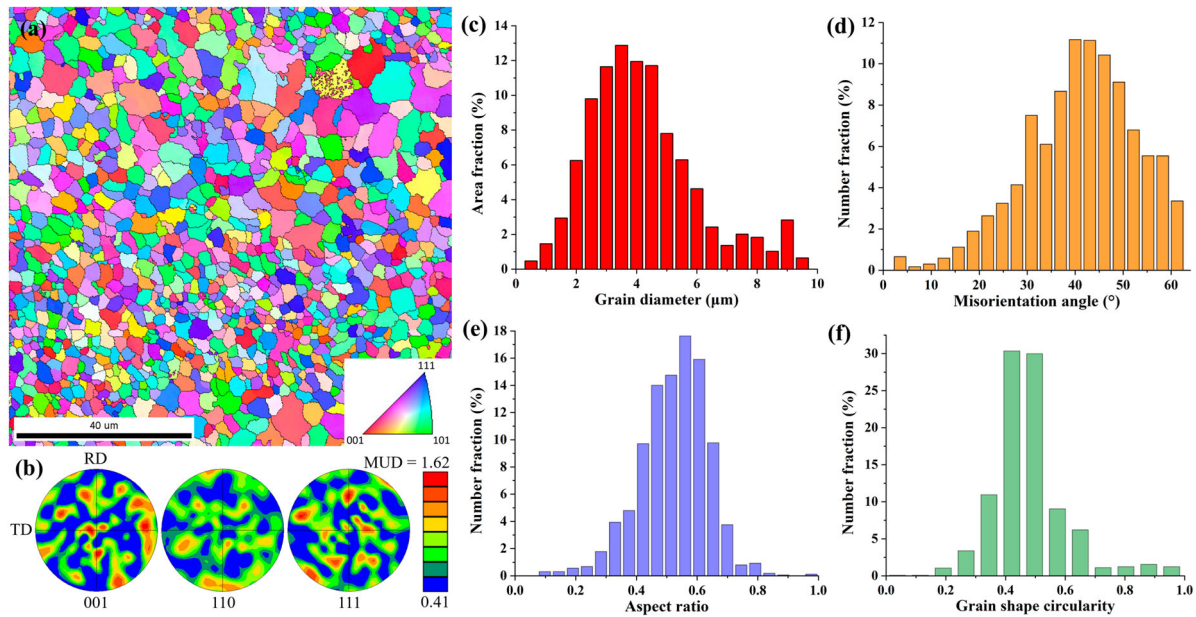


Figure 4. (a) EBSD IPF color map showing the grain structure and (b) pole figures showing the texture, (c) grain size distribution, (d) grain boundary misorientation distribution, (e) grain aspect ratio distribution, and (f) statistics of the grain shape circularity in the SLM-processed HEA-Cr₃C₂ sample. RD is the sample building direction.

To identify the phase structure in the as-fabricated HEA-Cr₃C₂ sample, TEM has been employed to investigate the material. The results are shown in Figure 5. Selected area diffraction patterns (SAED) and dark-field (DF) images taken from a (100) superlattice spot from grain interior or matrix area show that the grain interior or matrix area contains both disordered BCC and ordered B2 phase, which should be the result of spinodal decomposition that may have occurred during solidification after SLM. Similar BCC + B2 microstructure has been observed in other Al-rich HEAs during solidification [34,35]. The GB belt structure is identified to be the FCC phase. HRTEM images and Fast Fourier Transformation (FFT) pattern on the matrix area and the GB belt structure further confirm that the matrix is BCC/B2 phase while the GB belt structure is the FCC phase; see Figure 5(h–l). Measurement on the HRTEM image of the matrix gives rise to an interplanar spacing of 2.04 Å in two orientations, which correspond to (011) and (110) crystal planes of the BCC/B2 phase. The GB belt structure, however, possesses interplanar spacing of 1.84 and 2.08 Å in two directions, which correspond to the (111) and (002) crystal planes of the FCC phase, respectively. The formation of the FCC phase along GBs should be the result of segregation of C and Cr, which caused depletion of Al in these regions (Table 1) and promoted BCC/B2 → FCC transformation at GBs during solidification. Moreover, according to Qin et al. [24], the addition of C to a HEA can increase electronegativity and thus the interatomic forces of the alloy, which would promote the formation of the FCC phase.

Here in the present study, the significant enrichment of C at GBs would definitely elevate the electronegativity and interatomic forces in these regions, which favors the BCC/B2 → FCC transformation. The presence of FCC belt/network structure along inter-dendritic regions and GBs would help impede grains from abnormal coarsening and coalescing during subsequent SLM processing or thermal cycling.

Within some grain interiors, some precipitates that are enriched in Cr and C (Table 1) can be observed occasionally (Figure 5(c)). These particles are identified to be Cr₇C₃ carbides based on the SAED obtained from them (Figure 5(f)). However, since the population of Cr₇C₃ carbides in the sample is highly limited, it is unlikely that the CET and grain refinement are the results of heterogeneous nucleation on the Cr₇C₃ carbides. Also, given that the melting point of Cr₃C₂ (1895°C) is higher than that of Cr₇C₃ particles (1766°C) and that no Cr₃C₂ was observed in the as-fabricated samples, it is reasonable to believe that the Cr₃C₂ particles have been fully melted during SLM and that the Cr₇C₃ carbides should have formed from the liquid during solidification. It is also noted that Cr₂₃C₆ (with a melting point of 1576°C) is usually formed in as-cast C-containing HEAs [24], while Cr₇C₃ possesses higher stability at high temperatures [25]. Thus, the presence of Cr₇C₃ instead of Cr₂₃C₆ in the current as-fabricated sample should be the result of extremely high cooling rates after SLM, which may have suppressed the transformation of Cr₇C₃ into Cr₂₃C₆.

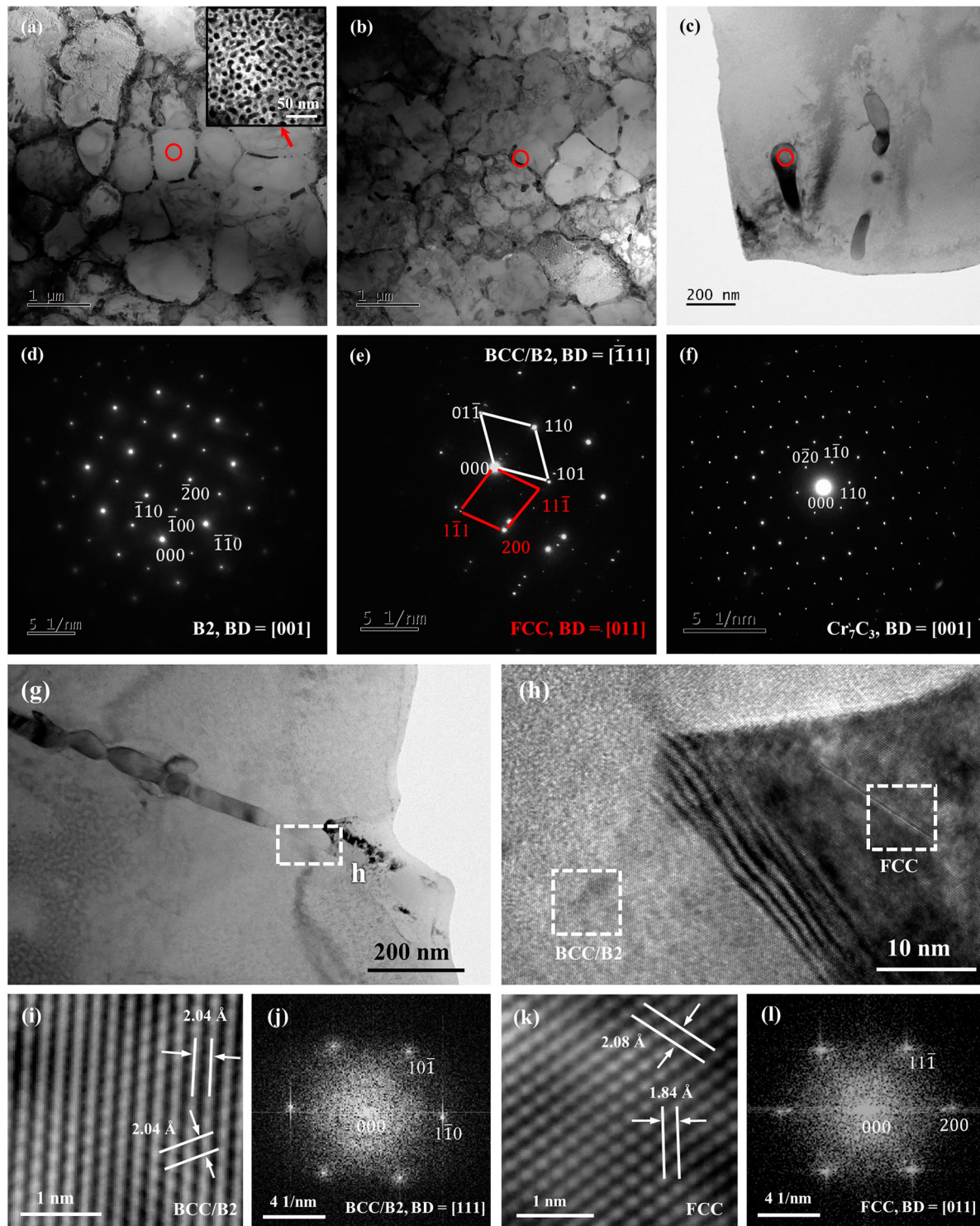


Figure 5. (a–c) Bright-field (BF) TEM micrographs showing fine grains, intergranular stripes, and particles inside grains of the HEA-Cr₃C₂ sample; The inset in (a) showing the DF TEM micrograph of the BCC/B2 matrix; Selected area diffraction patterns of (d) the BCC/B2 matrix, (e) the FCC phase and (f) Cr₇C₃ carbide indicated by the red circles shown in (a), (b) and (c), respectively; (g) BF TEM micrograph and (h) HRTEM image of the interface between the matrix and an intergranular belt structure; HRTEM micrographs and FFT patterns of (i and j) the B2 matrix and (k and l) an FCC belt structure indicated by the two square frames in (h), respectively.

Table 2 shows the microhardness of the SLM-processed HEA and HEA-Cr₃C₂ samples. The former shows a microhardness around 560 HV. With the

addition of Cr₃C₂, the microhardness has been remarkably improved. Thus, those regions containing fine dendritic grains show a microhardness of 590 HV, while

Table 2. Microhardness of the SLM-processed HEA and HEA-Cr₃C₂ samples.

Sample	Region	Hardness (HV)
HEA	Matrix-large columnar grains	559 ± 6
HEA-Cr ₃ C ₂	Fine dendritic grains	590 ± 6
	Coarse equiaxed grains	612 ± 9

the inter-layer regions with relatively larger equiaxed grains show a microhardness of 612 HV. The improvement in microhardness with the addition of Cr₃C₂ could be attributed to the refined grain structure and the solid solution of interstitial C in the matrix (Table 1). That those regions containing larger equiaxed grains in the SLM-processed HEA-Cr₃C₂ sample show higher microhardness than the regions consisting of finer dendritic grains could be because the latter contain not only FCC along GBs but also FCC along inter-dendritic regions, which should have softened the regions.

4. Conclusions

The addition of 5 at% Cr₃C₂ into AlCoCrFeNi alloy through SLM has led to full CET and significant grain refinement, which resulted in improvement in microhardness. The partitioning of Cr and C to the liquid ahead of the solidification front, which may have generated significant constitutional supercooling in the liquid, should have been responsible for the full CET. The segregation of Cr and C at GBs also led to a bimodal microstructure in the as-fabricated sample, i.e. the development of BCC/B2 grain interior and GB FCC belt structure/network. The present work indicates that by selecting a proper type of additive powder could a full CET be achieved in an AM-processed alloy.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

The data that support the findings of this study are available from the corresponding author upon request.

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