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ORIGINAL REPORTS



Unravelling the origin of multiple cracking in an additively manufactured Haynes 230

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ABSTRACT

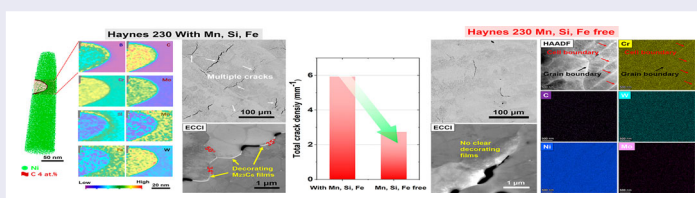
In this work, by using multi-scale characterizations from electron channeling contrast imaging (ECCI) to atom probe tomography (APT), we directly evidenced that the massive cracking events in the selective-laser-melted (SLMed) Haynes 230 superalloy are due to the continuous decoration of an $M_{23}C_6$ -type thin film at grain boundaries. The high-melting-point nature of the carbide rules out the possibility of liquidation cracking, while the long and straight film surface facilitates stress-induced solid-state cracking. Impurities, Si, Mn and Fe, greatly enhance the cracking susceptibility despite the interesting fact that they are strongly depleted from the carbide.

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Ni-based superalloy; additive manufacturing; cracking; elemental segregation; atom probe tomography



IMPACT STATEMENT

The massive cracking events in the selective-laser-melted Haynes 230 superalloy are due to the continuous decoration of an $M_{23}C_6$ film at grain boundaries, regardless of the detailed cracking modes.

1. Introduction

The rapid advancement of metal-based additive manufacturing (AM) technology has caused huge impacts on producing Ni-based superalloy parts in recent years [1–6]. This is because the AM process features near-net & high-freedom shaping, which hits the exact demand in jet engines or gas turbines. However, the Achilles heel of the widespread equipping of AM in the superalloy field mainly lies in the easy occurrence of cracking [7–13]. Two major cracking mechanisms have been proposed in the past decades referring to the case of welding. The first is hot-cracking, including solidification and liquidation cracking that originate from the reservation/formation of localized liquidation regions,

e.g. low-melting-point borides or eutectic phases along grain boundaries (GBs) [8,14,15]. The second is solid-state cracking, which occurs in pure solid-state and is driven by the accumulation of internal thermal stresses [7,16,17].

Actually, these cracks often appear simultaneously in the AMed superalloys and are hence believed closely related to each other [16,18]. One proposal is that the hot cracks developed during solidification or early thermal cycles can act as nuclei for the later solid-state cracks [7,18]. This is probably true since stress can be concentrated easily around pre-existed crack tips. However, direct proof is lacking while a clear picture depicting different cracking paths is still missing. A scientific question

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then arises: can there be a single root for multiple cracks in a certain AMed superalloy?

To address the concern, in this work we built specimens of Haynes 230, a classical solid-solution-strengthened Ni-based superalloy [19] via selective laser melting (SLM). Surprisingly we unravelled the continuous decoration of an $M_{23}C_6$ carbide film with a high-melting point at GBs that leads to most cracking events regardless of the specific cracking mode. Particular interest was paid to the segregation of impurities, i.e. Mn, Si and Fe, with the help of atom probe tomography. We believe that these findings will shed light on the future design of AM-suitable solid-solution-strengthened superalloys.

2. Materials and methods

The pre-alloyed Haynes 230 powder was produced using vacuum argon-protected gas atomization, with details described elsewhere [20]. SLM processing was conducted on an SLM 125HL machine (SLM Solutions GmbH, Germany) equipped with a 400 W fibre laser (IPG) under an argon atmosphere. The processing parameters are a laser power of 160 W, a scanning speed of 1000 mm/s, a hatching space of 90 μm , a layer thickness of 30 μm , a scanning rotation angle of 67° and a baseplate temperature of 200°C. The standard composition range and also the measured chemical composition of the SLMed sample via inductively coupled plasma (ICP) are listed in Table 1. Overall cracking morphology was revealed by scanning electron microscopy (SEM, Helios Nanolab G3 UC) under the second electron (SE) imaging mode and the electron-channelling-contrast (ECC) imaging mode. Electron backscattered diffraction (EBSD) was also correlatively carried out on a Zeiss GeminiSEM 460 machine. Microstructure details were then probed using transmission electron microscopy (TEM, FEI Titan G2 60-300) with an objective aberration corrector operated at 300 kV. SEM/EBSD samples were prepared by minor polishing, using a 50 nm scale SiO_2 suspension. TEM samples were twin-jet electro-polished in a solution of 10% HClO_4 + 90% $\text{C}_2\text{H}_6\text{O}$ at a direct voltage of 30 V. Atom probe tomography (APT) was carried out on a LEAP 5000XR from Cameca Instruments Inc., under a laser pulsing mode with a pulse repetition rate of 125 kHz and a pulse energy of 60 pJ. The base specimen temperature was set at 60 K, and the target detection rate was kept at 0.7%. APT needle specimens were prepared following the standard lift-out protocol in a dual-beam SEM/focused-ion-beam (FIB) instrument (Helios Nanolab 600i).

3. Results and discussion

Figure 1a gives a cross-sectional view of the as-built microstructure. Dense distribution of cracks appears, with cracking lengths ranging from several to hundreds of microns. After carefully screening more than 200 different cracks we find that firstly, cracking occurs exclusively along normal high-angle GBs (nearly 98% in number fraction). And secondly, as shown in Figure 1b both hot cracks (to be more specific, solidification cracks) with an exposed dendritic arm on crack faces and solid-state cracks that are straight (many with sharp corners) exist here and there. Notice that as plotted in Figure 1b more than half of the checked cracks are not able to distinguish precisely under the commonly applied morphological criterion. Correlative ECCI-EBSD observations on several of those cracks are provided in Figures 1c–f, respectively. We number the crack in Figures 1c and d as crack 1, and the misorientation across the cracked GB falls in the range of 30–40° (measured from the inverse pole figure (IPF) in Figure 1(d)). By taking a closer view of cracks 1 and 3 in Figures 1h and i, respectively, we observe that both the cracks are discrete and consist of short pieces of cracks, suggesting that they are not fully opened/developed. These premature cracks allow us to collect real information about the crack-sensitive GBs right before they split into large cracks. Surprisingly, abundant linear features with the brightest contrast are found connecting those isolated short cracks. Even the uncracked low-angle GB ahead of the crack tip (the 14° one in Figure 1i) is continuously decorated by the brightest feature. These observations indicate that the brightest feature, probably a new phase, preliminarily covers almost all cracked GBs regardless of the misorientation value and cracking mode. This resembles the case of the so-called liquid film with low-melting-point mentioned before in the literature [8,16,21].

The film-like phase is further characterized by TEM in Figure 2. We prepared a site-specific lamella sample across a cracked GB, as described in Supplementary material Figure S1. The overall TEM bright field imaging of the lamella is given in Figure 2a. The width of the subject crack separating the two grains (containing columnar dislocation substructures) is, to some degree, expanded during the FIB-milling process. The film-like phase, as pointed by the yellow arrow, is embedded ahead of the crack, near the sample surface. Figure 2b is a closer view showing the film-like phase, while Figure 2c gives the high-resolution TEM image centred on the phase boundary. The comparison of the fast Fourier transformation (FFT) in the insets of Figure 2c indicates that

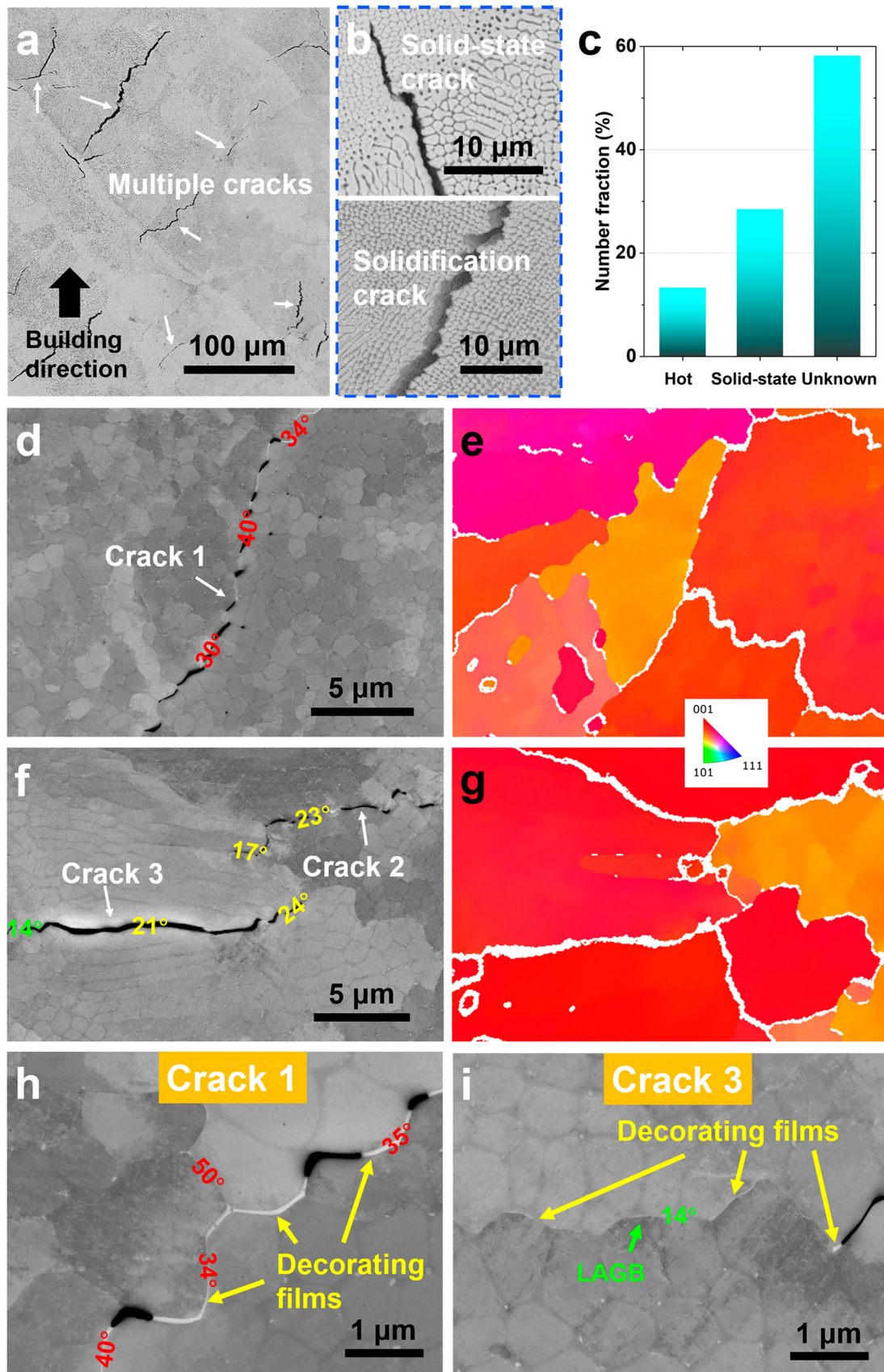


Figure 1. Crack identifications in the SLMed Haynes 230 superalloy: (a) Low-magnification image showing the overall cracking status. (b) High-magnification images showing examples of a solid-state crack and a hot-crack (solidification crack). (c) Columnar charge showing the number fractions of the identified cracks and those unresolved cracks. (d, e) and (f, g) Correlated ECC-EBSD images showing two cracked regions with three cracks numbered 1, 2 and 3; (h, i) High-magnification ECC images showing the microstructures centred on the crack 1 and 3, respectively, where film-like features are clearly observed.

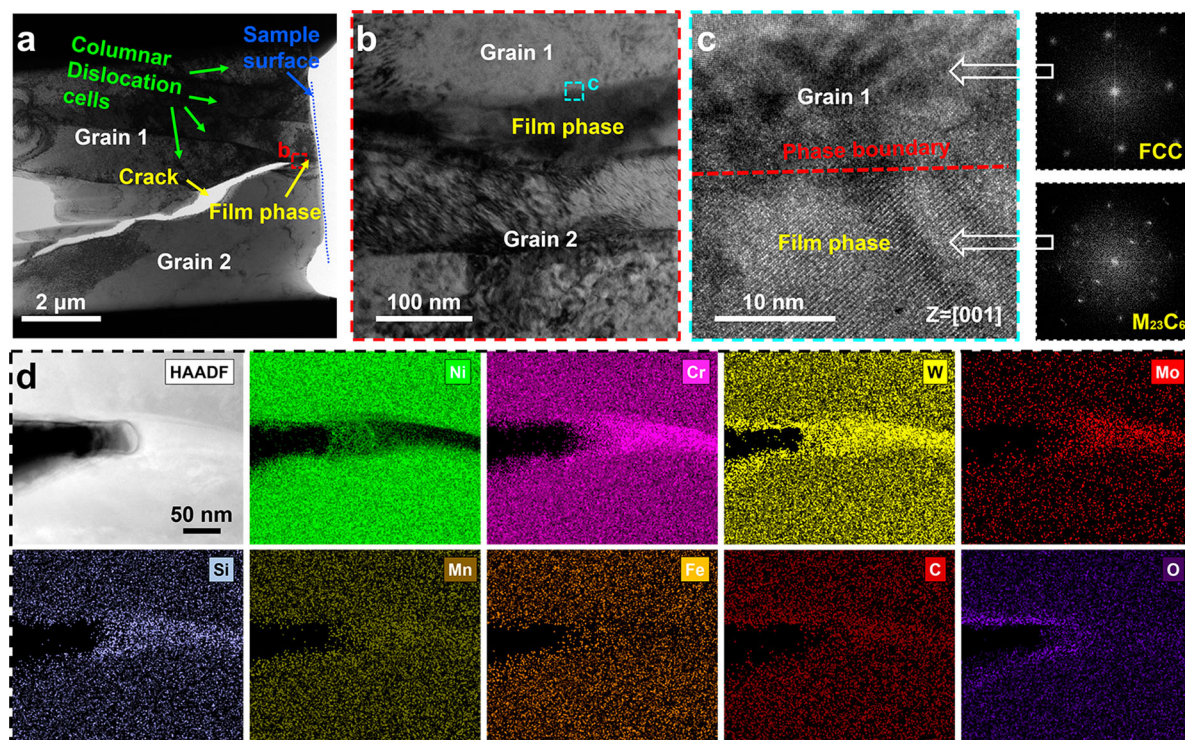


Figure 2. TEM observations on the film-like phase: Bright field images showing (a) the overall view of the lift-out lamella sample and (b) the film-like phase enclosed to the crack. (c) High-resolution TEM image centred on the interface of the film-like phase and the matrix, with insets showing their FFT patterns, respectively. (d) STEM-HAADF image and the corresponding elemental maps centred on the crack tip and the carbide film.

it is an $M_{23}C_6$ carbide that maintains a near-coherent $\{100\}$ interface with the face-centred cubic (fcc) solid-solution matrix [22]. Figure 2d shows the scanning transmission electron microscopy (STEM) high-angle annular dark field (HAADF) image on the $M_{23}C_6$ carbide and the abutted crack tip. The elemental distribution maps indicate that the carbide is enriched highly in Cr, W and Mo but slightly in Si and C. For Si/C the enrichment intensity might not be correctly reflected due to the limited EDS accuracy on light-weight elements. In addition, only Cr, W and O are found co-segregating at the crack inner face with a thickness of only 10 nm. Combining with the fact that no obvious O enrichment is detected at the crack tip carbide position (which is also in line with the APT results in Figure 3), it is supposed that such oxidation occurs along the crack channel right after the crack extension during SLM, hence probably won't generate key influences on the crack onset.

To reveal the chemistry (in particular Si, C and B) of the $M_{23}C_6$ carbide more precisely, we carried out APT measurements, and the results are shown in Figure 3. Figure 3a outlines the carbide-matrix interface at a 4.0 at.% C iso-composition surface. Figure 3b lists all two-dimensional (2D) elemental maps from the

orange-coloured region of interest. The carbide enriches in C, B, Cr, Mo, W, in accordance with the TEM-EDS mapping results in Figure 2d except for Si. In the APT dataset Si is enriched surrounding the carbide. It is believed that the 2D overlapping of the Si-rich nano-layer with the carbide volume leads to the artifact projected by TEM. The composition of the carbide is Cr45.1-Ni17.8-C13.7-W8.3-Mo8.2-B3.0-Si1.9-other (at.%) according to the 1D composition profiles in Figures 3c and d, along the black arrow in Figure 3b. The Cr level in the carbide is detected at around 45 at.%, in line with the reported $M_{23}C_6$ carbide in the literature [20,23,24]. In addition, B is probed similarly segregated towards the phase boundary in the carbide side, indicating continuous absorbance of B and repulsion of Si across the interface during the growth of the carbide.

The chemistry on low-angle GBs is also probed using APT and the results are given in Supplementary material Figure S2. The elemental segregation intensity is reasonably similar, and more likely stronger at high-angle GBs due to the higher local free energy and thus the higher segregation driving force [25]. Therefore, by comparing GBs and carbide films, the apparent segregation of Cr, C and B will facilitate the formation of the $M_{23}C_6$ carbide by reducing the nucleation barrier, and eventually construct

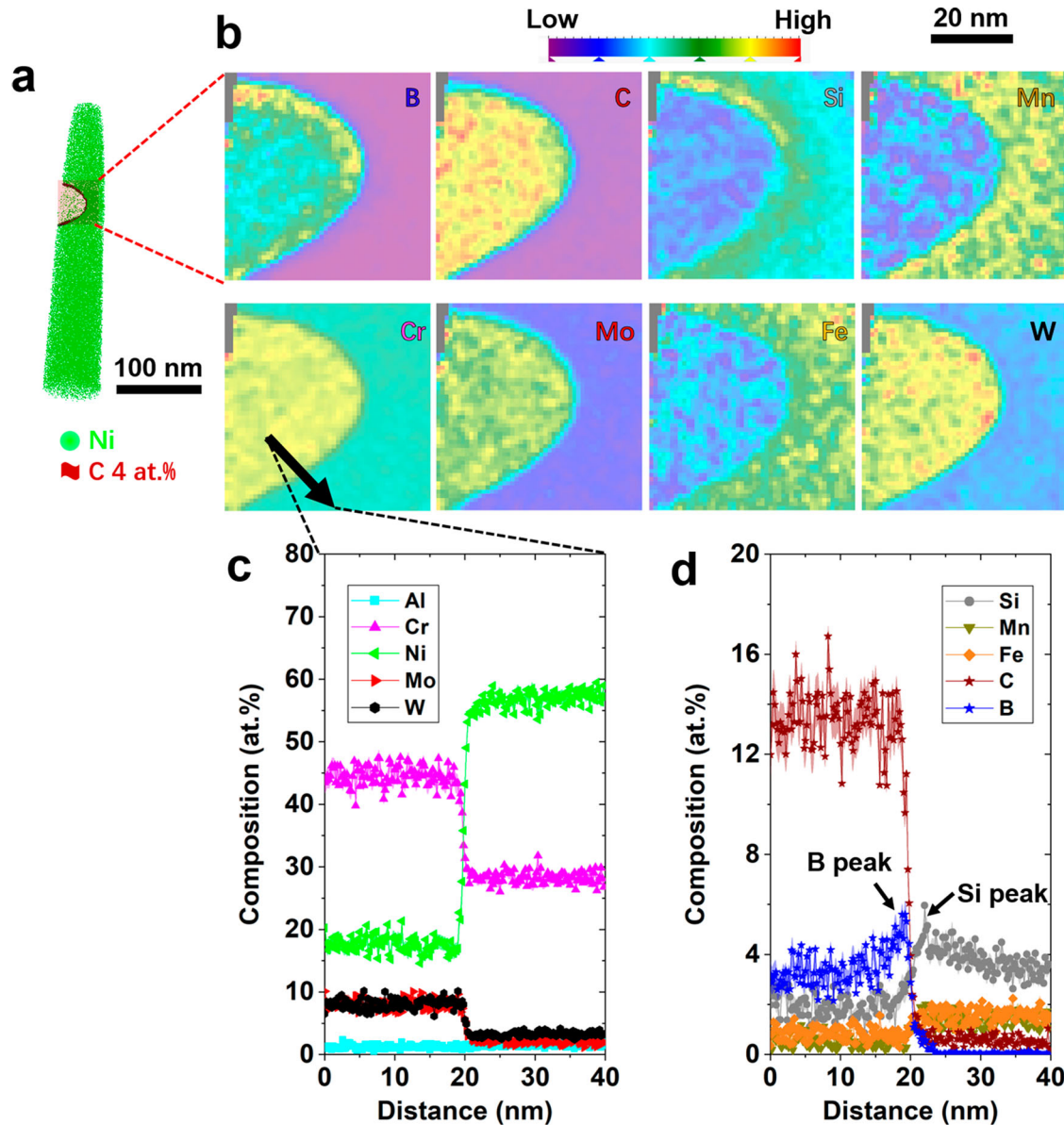


Figure 3. Atom probe tomography on the $M_{23}C_6$ carbide: (a) APT reconstruction with a 4.0 at.% C iso-composition surface embedded in Ni atoms showing the presence of the carbide. (b) 2D elemental distribution maps in the orange region of interest. (c, d) 1D composition profiles along the black arrow in (b).

it as the principal element. Mo and W are also strong carbide stabilizers [20,23], and they indeed enrich the carbide despite no clear segregation at GBs. This is reasonable considering their 1~2 orders of magnitude lower diffusion kinetics than the solvent Ni. However, considering the key roles played by the five elements on the temperature capability for a polycrystalline solid-solution-strengthened superalloy, it is not preferred to exclude or even reduce their amount in the alloying philosophy. Among the left, Mn, Si and Fe are found strongly repelled from the carbide, behaving just like carbide-destabilizers. This is quite interesting since that Mn and Si are often reported as crack promoters [26–28]. In particular, Si is

evidenced as a significant stabilizer for yet another type of carbide, M_6C , and with a high tendency to enrich along multiple interfaces (as depicted in Figure 3d and Figure S2d) [29–31]. Although Fe is seldom seen in the discussion on forming carbides in superalloys, its interaction with Cr to construct $(Fe/Cr)_{23}C_6$ in steels is well acknowledged [32,33]. To figure out whether the formation of the carbide film during SLM is related and can be enhanced by the alloying of the three elements, we carried out a combined experimental-thermo-calc investigation on an Mn, Si, Fe-free counterpart (measured composition listed in Table 1), and the comparison to the current Mn, Si, Fe-containing Haynes 230 is given in Figure 4.

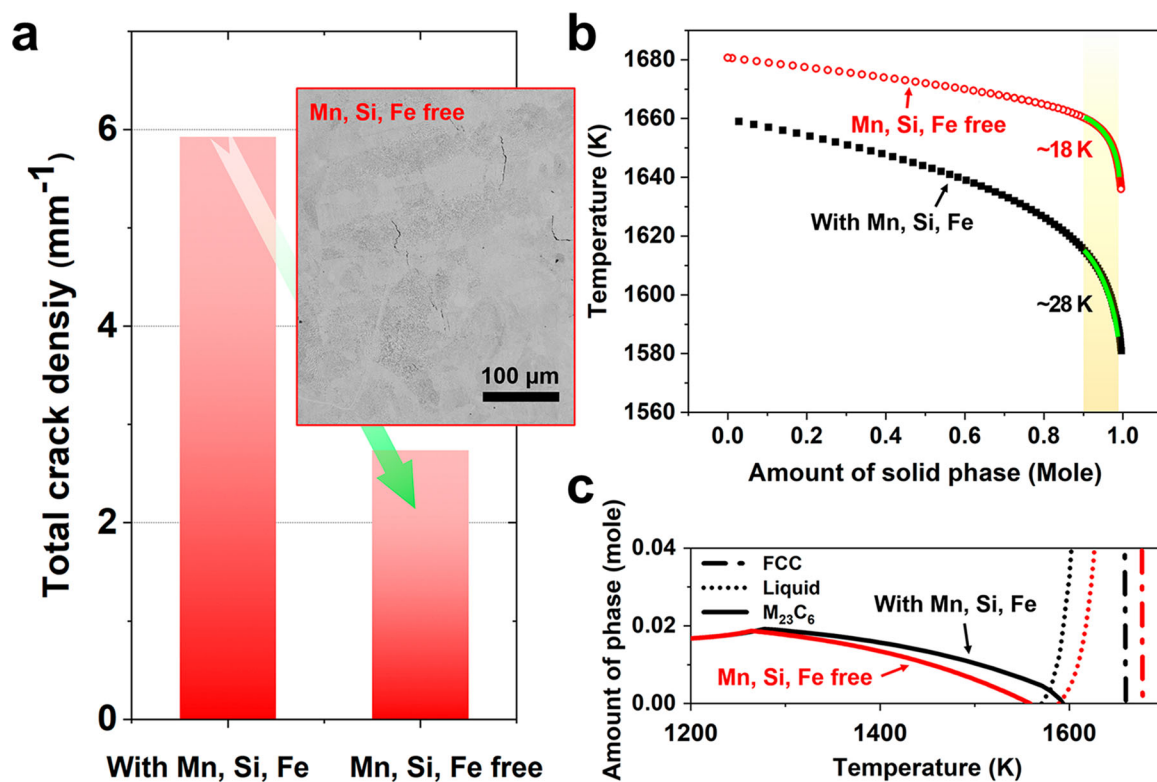


Figure 4. Combined experimental-thermo-calc investigation on the Mn, Si, Fe-free counterpart and the current Mn, Si, Fe-containing Haynes 230: (a) Columnar chart showing the measured crack density, with an inset showing the overall crack status in the Mn, Si, Fe-free counterpart; Thermo-calc simulation showing (b) the solidification path based on the Scheil-Gulliver assumption and (c) the equilibrium phase curve focusing on the $M_{23}C_6$, fcc and liquid phases, using the TTNi8 database.

Table 1. Standard composition range and also measured composition of the SLMed Haynes 230 superalloy (the current one with, and the modified one without Si, Mn, Fe, all in wt.%).

Haynes 230	C	B	Si	Mn	Fe	Mo	W	Cr	Al	Ni
Standard range	0.05–0.15	≤ 0.015	≤ 0.75	≤ 1.00	≤ 3.00	1.00–3.00	13.00–15.00	20.00–24.00	0.20–0.50	–
Current with Si, Mn, Fe	0.0658	0.0056	0.391	0.488	1.778	2.034	12.987	20.099	0.327	–
Modified Si, Mn, Fe-free	0.0703	0.0041	0.022	< 0.005	0.049	1.955	13.569	21.989	0.432	–

Figure 4a shows the low-magnification image of the microstructure of the SLMed Mn, Si, Fe-free counterpart, where much fewer cracks appear. The measured total crack density drops substantially from 5.93 to 2.74 mm^{-1} , indicating a lowered cracking risk by removing Mn, Si and Fe. Supplementary material Figure S3 (ECCI) and S4 (STEM) further confirm barely no similar film-like phase covering cracked GBs in this counterpart. The solutes (e.g. Cr) segregation at the dense cell boundaries, as shown in Figure S4, suggest that the reduced tendency to form $M_{23}C_6$ along GBs might be due to the averaged segregation intensity of certain elements towards multiple boundaries rather than GBs only, when missing Mn, Si and Fe in the system. Figure 4b shows the thermo-calc result on the solidification path from the Scheil-Gulliver assumption, using the TTNi8 database. We highlight the critical temperature window in the last solidification stage, ΔT_{CRI} , where the fraction of solid changes from 0.9 to 0.99 (a yellow coloured region in

Figure 4b) [16,34]. The larger the ΔT_{CRI} , the higher strain and strain rate will be generated during the very terminal stage of solidification, resulting in higher solidification cracking sensitivity. Here for the Mn, Si, Fe-free one, the ΔT_{CRI} is only around half of that for the Mn, Si, Fe-containing one, also suggesting a reduced solidification cracking sensitivity, in line with Figure 4a. Figure 4c then shows the thermo-calc result on the equilibrium phase composition, including only the $M_{23}C_6$, fcc solid solution and liquid phases in the near-liquidation temperature range. It is shown that the upper-temperature limit of equilibrium $M_{23}C_6$ turns higher when including Mn, Si and Fe in Haynes 230 (black solid line vs. red solid line). This suggests that Mn, Si and Fe stabilize the $M_{23}C_6$ phase anyway although they do not directly construct the crystalline lattice of $M_{23}C_6$.

Across the observations above, we can address the same immediate structural origin for most cracks in the SLMed Haynes 230 as the formation of the $M_{23}C_6$ thin

films along GBs. This seems in line with the reported hot-cracking mechanism being the tearing of the weak liquid film [7,8]. Further considering a very important fact that the carbide film itself is not of low-melting-point, as pointed out in Figure 4c, the hot tearing process may not follow the liquidation cracking mechanism. On the one hand, although B segregation at GBs indeed exists in our case, it remains insufficient to form those low-melting-point borides, as illustrated by Kontis et al. [21] and Després et al. [35]. Instead, the segregated B atoms flow into the carbide at a low concentration of ~ 3 at.%. On the other hand, according to Guo et al. [36] and Zhang et al. [37], $M_{23}C_6$ can still correlate to liquidation cracking only if the carbide forms in a eutectic way, creating a low-melting-point eutectic regime along GBs. What's more, no liquidation trace is observed near or ahead of the cracked GBs. Thereby, we exclude the occurrence of liquidation cracking in the current SLMed Haynes 230.

As a new root cause for cracking in at least the current Haynes 230 superalloy, we propose the following cracking sequence associated with $M_{23}C_6$ films along GBs. During the terminal stage of solidification, C, B and Cr, etc. are repelled from the solidified dendrites to the surrounding liquids [36]. The local enrichment of these carbide stabilizers accelerates the nucleation of $M_{23}C_6$ directly from the liquids, as shown in Figure 4c where the upper-temperature limit of the equilibrium $M_{23}C_6$ is nearly 20 degrees higher than the end-of-solidification temperature. Considering the extreme case where the $M_{23}C_6$ phase forms within the interdendritic liquids, its film-like presence will certainly block the liquid feeding hence resulting in solidification cracking nearby. Such blocking effect is reasonably stronger than that from discrete tiny spherical MC carbide or Re-enriched precipitates as claimed in the literature [7,14]. Nevertheless, the identification of a minor number of solidification cracks supports the conjecture above. Alternatively, in a more reasonable case where the $M_{23}C_6$ phase grows and coarsens continuously along the GBs during the following thermal cycles, the bonding of the carbide/fcc matrix interface is likely not strong, which could be further weakened by the Si-enriched nano-layer surrounding the carbide. The straight and large film surfaces also lower the tolerance for strain and stress partitioning and once the micro-voids initiate at the surface, they coalesce and extend fast into a long crack. This suggests that the stress-driven solid-state crack can occur much more easily along the $M_{23}C_6$ films, in line with the identified larger number fraction of solid-state cracks compared to hot cracks. In this context, we suppose that most of the unresolved cracks are also of solid-state type.

4. Conclusion

In this study, we reported that originating from the high-melting-point nature, the formation of the $M_{23}C_6$ carbide films along GBs directly leads to massive cracking events in a solid-solution-strengthened Ni-based superalloy, Haynes 230. This is seen as a general cracking origin for this specific superalloy, regardless of the GB misorientation and the specific cracking mode. By using APT, we evidenced that the segregation of Cr, C and B fundamentally triggers the nucleation of the carbide. Although the impurities, Mn, Si and Fe, are repelled from the carbide, their removal successfully suppresses cracking during SLM. This is because firstly, they enhance the solidification-cracking sensitivity by both the enlarged ΔT_{CRI} and the stabilized $M_{23}C_6$ phase over the liquidus line; and secondly, they promote the growth of the $M_{23}C_6$ film along GBs, which concentrates stress and causes solid-state tearing along the straight weak interfaces.

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Disclosure statement

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

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