The effect of heat treatment on the metallurgical properties of FeCo alloy

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Abstract. Fe-Co alloys, in particular B2 FeCo, are considered attractive soft materials due to their superior properties, particularly their high saturation magnetization and mechanical strength in applications such as electricity powerlines, aerospace and automotive industries. Despite its application potential, B2 FeCo alloy is characterized by limited ductility at room temperature. In this study, the effect of heat treatment on the constituent phases, microstructures and micro-Vickers hardness of FeCo alloy is investigated. Phase and microstructural evolution were tracked using X-ray diffractometer (XRD) and optical microscope (OM), respectively. The X-ray analysis of the as-cast condition (AC) and furnace cooling (FC) displayed α'-B2 phase peaks with traces of α phase while ice-water quenched (WQ) at 750 °C and 1000 °C showed only α phase. OM of WQ at 750 °C samples showed wide equiaxed α' grains, while the AC, WQ (1000 °C) and FC demonstrated finer grains inside and around the grain boundaries. Other metallurgical analyses carried out include micro-Vicker hardness tests, from which the WQ (1200 °C) sample demonstrated the highest hardness value as compared to both AC and FC samples.

1 Introduction

FeCo alloys are considered as attractive soft magnet materials in various engineering applications owing to their superior properties, particularly their high saturation magnetization, high melting point and high Curie temperature (T_C) [1]. A near-equiatomic FeCo alloy consists of a disordered face-centered cubic (FCC) γ phase at temperatures above 985 °C and disordered body-centered cubic (BCC) α phase at temperatures between 985 and 730 °C as shown in Figure 1 [2]. Furthermore, the BCC solid solution phase transforms to an ordered B2 α′ structure at temperatures below 730 °C. It is this intermetallic compound that exhibits superior magnetic properties such as high T_C, good permeability and high saturation magnetization, which renders this material suitable candidate for applications that requires high flux densities, i.e. efficient power transmission lines as a result of low core losses [3]. However, due to its brittle character, the B2 FeCo phase suffers from low level of ductility at room temperature, which makes this alloy difficult to form during cold-working process [4].

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In attempts to improve the ductility of the FeCo alloy, it has been reported that increasing Co content [4] and assigning wavy slip at grain boundaries by partial ordering [5] enhances ductility. It is also reported that low coercivity and core losses can be obtained through subjecting the FeCo alloy to high heat-treatment temperatures followed subsequent rapid cooling leading to grain refinement [3]. As opposed to single ordered α′ phase formed through cooling under equilibrium condition, rapid cooling may lead to coexistence of disordered α and ordered α′ phases, leading to increased ductility due to the presence of disordered BCC phase. Thus, a trade-off between strength and ductility at room temperature can be achieved by tailoring a suitable heat-treatment cycle. As a result, the optimisation of metallurgical properties through heat treatment processes remains critical area of research. Heat treatment can significantly influence the microstructure and phase constituents of FeCo alloys, subsequently affecting the hardness, magnetism and overall mechanical performance. Understanding the effects of treat treatment is essential for enhancing the applicability of this alloy in advanced technological applications.

Previously, in a related study on FeCo-based alloys, it was demonstrated that it is important to anneal at high temperatures, specifically 1000 °C, in altering the phase composition and microhardness of 20Co-Cr-Fe-Ni alloys [6]. It was found that the microstructures consist predominantly of BCC and FCC phases, with the stability of theses phases highly temperature dependent. The transition from FCC to BCC was found to occur below 700 °C, suggesting that heat treatment parameters must be closely controlled to maintain desired properties. In another study, the thermal treat treatment modulated on rapidly quenched FeCo-based alloys was investigated [7]. The analyses of magnetization behaviour were conducted at 667 °C. It was noted that the transition from amorphous to crystalline phases significantly alters the magnetic saturation characteristics. It was also observed that an increase in atomic diffusion during heat treatment favours the development of hard magnetic phases that contribute positively to magnetisation thereby improving the overall magnetic performance of the FeCo alloy compositions. The emerging of these hard magnetic phases under controlled thermal condition highlights the critical role of heat treatment in tailoring the magnetic properties of FeCo alloys, a factor crucial for optimizing performance in applications such as magnetic sensors and actuators. Other work explored heat treatments of FeCo in the context of new manufacturing processes like additive manufacturing using laser powder bed fusion, where annealing was found to improve properties like coercivity and power loss [8]. It was also found that a 2-hour normalization heat treatment followed by a 4-hour primary heat treatment at 1123 K and slow cooling to room temperature produced a recrystallized microstructure with mainly equiaxed grains with an average size of up to 61 μm and a fully ordered B2 structure, characterized by a high degree of order.

Considering the importance of heat treatment in the fabrication process of binary FeCo alloys, the current study examines the effect of heat treatment on the crystal structure, microstructure and mechanical properties.

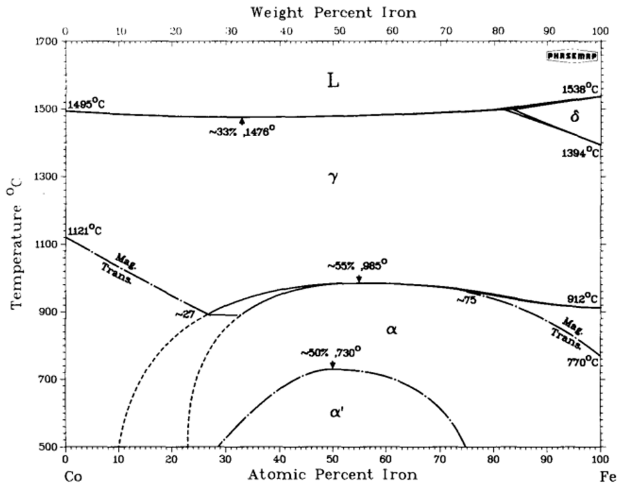


Fig. 1. FeCo binary phase diagram [2].

2 Materials and methods

2.1 Materials preparation

A 50 g binary FeCo alloy button was produced using a state-of-the-art button arc melting technique. During the melting process, commercially available high-purity iron (Fe) and cobalt (Co) metallic powders were compacted and melted in an ultrasonic arc furnace, equipped with a tungsten electrode and a water-cooled copper hearth. The melting process was conducted under a controlled argon atmosphere to prevent oxidation, and the ingot was flipped and re-melted three times to achieve homogeneity. This approach ensured uniform distribution of alloying elements throughout the ingot. Five samples were sectioned, from which one sample was kept in the as-cast condition whereas the other four samples were subjected to solutionizing treatment in a muffle furnace at 1200 °C for 1 hour followed by furnace cooling (FC) and subsequent quenching from 1200 °C, 1000 °C and 750 °C into ice-water. The resulting samples were mounted, ground and polished to a 1µm surface finish for metallurgical characterisation.

2.2 Microstructural and phase evolution

The X-ray diffractometer (XRD) and optical microscope (OM) were used to analyse the phases present and microstructure, respectively, of the 5 samples. XRD analysis was carried out using Bruker 8 Discovery XRD machine running over a 2θ range of 20º to 100º at a scanning rate of 0.5 º/s with a step size of 0.02º using a Co Kα radiation source. Following the metallographic preparation methods, OM samples were prepared and etched for 30 seconds in a solution containing 100 mL distilled water, 100 mL hydrochloric acid, 200 mL methanol, 5 mL nitric acid, 7 g ferric chloride and 2 g cupric chloride. Immediately after etching, the samples were cleaned with alcohol, followed by water. An optical microscope (OM) was used to image the samples using a 200 µm scaling bar for microstructure analysis.

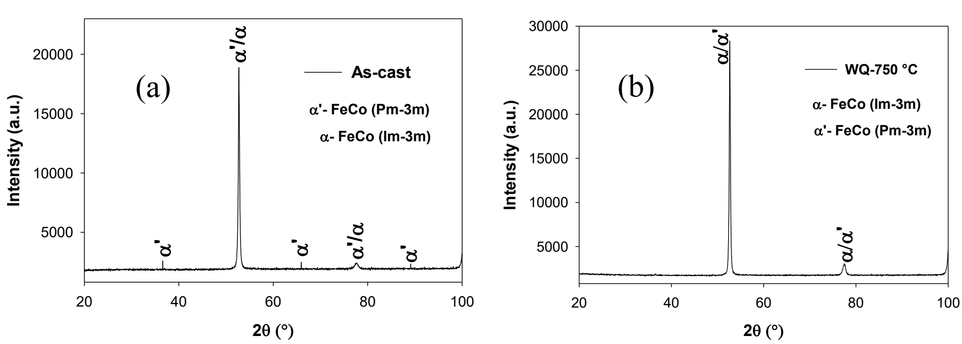
2.3 Hardness test

Zwick Roell Vickers hardness testers were used to perform micro-Vickers hardness tests to assess material strength. The test was conducted in accordance with ASTM E E384-22 (Standard Test Method for Microindentation Hardness of Materials). A micro hardness test was conducted on each of the 5 samples, with 5 indents per specimen made and measured microscopically according to the H_V scale. For each sample, an indentation was made with a load of 500gf for 10 s, followed by two diagonals that were measured microscopically. The average of the diagonals was then calculated.

3 Results and discussion

3.1 X-ray diffractometer

The samples subjected to various cooling rates were analysed using XRD to identify present constituent phases. Figure 2 depicts the XRD patterns of FeCo alloy samples in as-cast (AC), water quenched (WQ) and furnace cooled (FC) conditions. There is clear evidence of the α-FeCo crystalline phase by main diffraction peak around 2θ = 52.8º and 2θ= 77.8º that is found for investigated samples. The presence of α′-FeCo phase at peaks 2θ= 36.6º (weak peak), 2θ= 52.8º (strong peak), 2θ= 65.9º (weak peak), 2θ= 77.8º (strong peak) and 2θ= 89.1º (weak peak). The AC sample exhibited peaks corresponding to the cubic α′ and α phases. This α′ phase is seen at peaks 2θ= 36.6º, 2θ= 52.8º, 2θ= 65.9º, 2θ= 77.8º and 2θ= 89.1º. This suggest that the AC consists of B2 phase as shown in Figure 2 (a). Furthermore, the Figure reveals the presence of a BCC structure in small amount, which is the most common crystal structure for FeCo alloy. Albaaji et al. reported similar results on as-cast sample [9]. It was also observed that the sample quenched from 750 ºC exhibits α phase only. The existence of the α phase peaks in this alloy indicate the quenching medium was sufficient enough to keep the α phase as it exists at that temperature. The XRD patterns of the WQ (1000 ºC) sample revealed the presence of α phase with peaks at 2θ = 52.8º and 2θ =77.8º. The structures which exists in this phase include FeCo and $FeCo_3$ with cobalt content of 50 and 75 at.%. The peak intensity at 2θ=77.8º was found to be high as compared to other cooling media which might be due to the high crystallinity of coarse grains. The formation of α phase peaks was attributed to the rapid cooling rate. Furthermore, the XRD patterns of the WQ (1200 ºC) sample revealed the presence of α′ (FeCo) phase at peaks between 2θ=36.6º, 2θ=52.8º, 2θ=65.9º, 2θ=77.8º and 2θ=89.1º, and α ($FeCo_3$) phase at peak 2θ=53.2º. When the sample was furnace cooled (at T= 1200 ºC), the XRD peaks illustrated α′ phase at 2θ= 36.6º, 2θ= 52.8º, 2θ= 65.9º, 2θ= 77.8º and 2θ=89.1º but the peaks at 2θ= 36.6º, 2θ= 65.9º and 2θ=89.1º were very weak. The intensity of α′ shows a similar intensity degree as the previous study although the heat treatment was performed at 1100 ºC using ball milling [10].



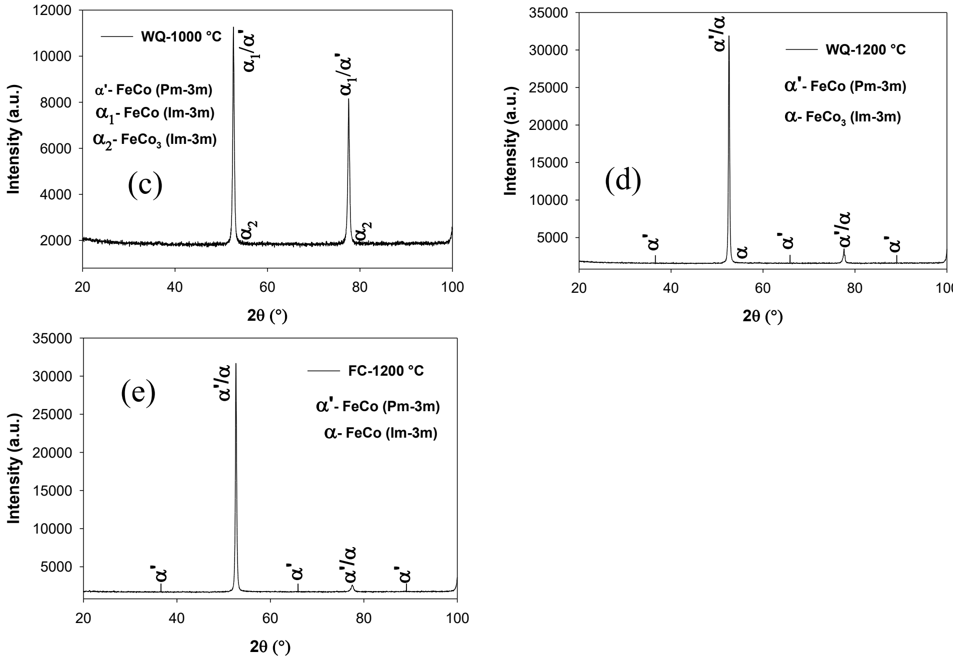


Fig. 2. X-ray diffraction of FeCo at different cooling types: (a) As-cast, (b) WQ at 750 °C, (c) WQ at 1000 °C, (d) WQ at 1200 °C and (e) FC at 1200 °C.

3.2 Optical microscope

Figure 3 shows an optical microscope (OM) image of FeCo alloy subjected to different cooling rates. The OM micrographs of AC sample in Figure 3 (a) were comprised of fine equiaxed grains of α'-B2 phase. Furthermore, the grain boundary in as-cast sample was found to be very fine, suggesting that the material could be harder. As can be seen in Figure 3 (b), WQ in 750 °C micrographs were characterized by coarse grains of equiaxed phase, whose orientation and sizes are different. This suggest that rapid cooling was sufficient to maintain the α phase which exist between 730 °C and 985 °C. In addition, the grain boundary in the WQ sample was found to be clear. Compared to 750 °C and AC samples, the WQ at 1000 °C sample micrographs in Figure 3 (c) have significantly finer grains inside and on boundaries between grains. This is attributed by grain pinning, where the movement of dislocations is hindered by fine precipitates within the grains which will result in high strength, elongation, and toughness in the material. In Figure 3 (d), the QW at 1200 °C sample showed finer grains than the WQ at 750 °C and 1000 °C; inside the grain there were different substructures. OM could not clearly identify substructures, but XRD patterns indicated they were associated with α' and α phases. The micrograph in Figure 3 (e) was composed of equiaxed coarse and fine grains of α′ phase around the grain boundaries and inside the grains. Differences in grain size in the samples can be attributed to the fact that the cooling medium between WQ samples and the diffusion in AC and FC samples. Due to the diffusion process in the cooling medium, the AC and FC samples showed similar structures. As compared to WQ and FC, AC sample with a little slow cooling rate displayed no-recrystallization due to differences in grain formation. As a result, finer grains formed because the cooling medium was too high to allow full recrystallization. Consequently, it was difficult to compare current work with literature since there has been little work reported with this process.

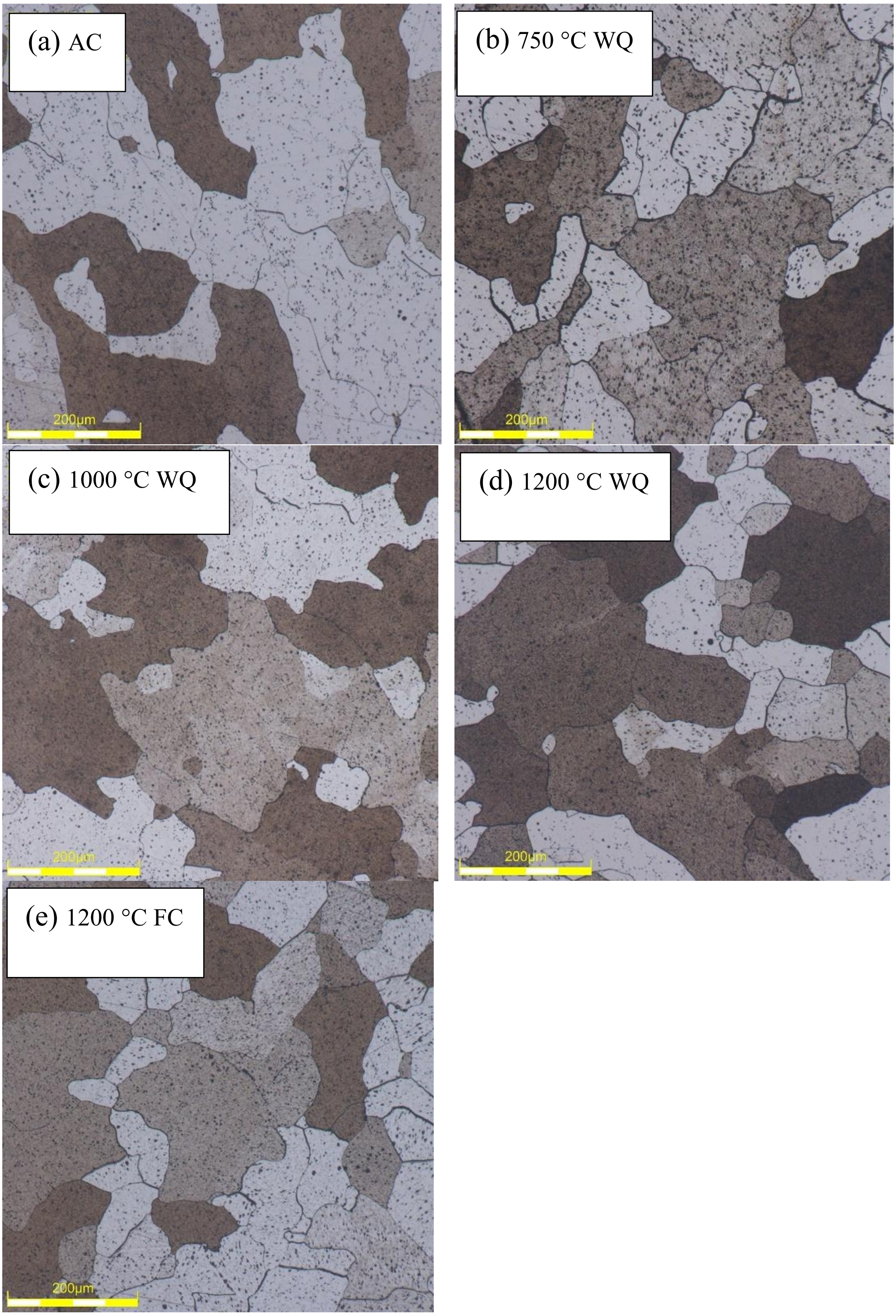


Fig. 3. Optical Micrographs of FeCo alloy in different cooling types (a) As-cast, (b) WQ at 750 °C, (c) WQ at 1000 °C, (d) WQ at 1200 °C and (e) FC from 1200 °C.

3.3 Micro-Vickers hardness

Figure 4 illustrate the Micro-Vickers hardness of FeCo at various cooling rates. Hardness measurement is a straightforward method to understand how different heat treatment conditions affect mechanical properties. Those samples that were cooled in a variety of cooling media showed the following hardness values: as-cast samples exhibited a hardness value of 213.50 $Hv_{0.5}$, ice-water quenched (WQ) samples at 750 °C had the lowest hardness of 185.94 $Hv_{0.5}$, WQ samples at 1000 °C showed a hardness value of 207.82 $Hv_{0.5}$, WQ samples at 1200 °C showed the highest hardness of 252.68 $Hv_{0.5}$, whereas furnace cooled samples (FC) were rated at 200.00 $Hv_{0.5}$. Possibly, the low hardness value for the quenched sample at 750 °C is the result of coarse grain size, which reduces the strength of the material further by allowing more dislocation motion as compared to finer grains, as shown in Figure 3. Due to the fast cooling or recrystallizing from a high temperature, the grains nucleated and grew, suggesting the sample is ductile. Moreover, it might be attributed to the high temperature phase (FeCo-Im-3m) which exists at that temperature. In comparison with other samples, WQ sample at 1200 °C has a high hardness. This may be due to higher volume fraction of α phases compared to the as-cast condition and FC samples with α′ phase existence. In Figure 4, the WQ sample has a higher hardness largely due to its fine grains and the high temperature phase compared to the AC and FC samples in Figure 3 (a) and (e). The fine grains in WQ at 1200 °C inhibit dislocation movement, resulting in increased hardness and strength, whereas coarse grains allow dislocation movement and decrease hardness. It is, however, noted that the hardness of as-cast samples is higher than that of WQ (750 °C and 1000 °C) and FC samples. FeCo alloy has previously been found to have a hardness between 170 H_V and 265 H_V [11], which is similar to the current study. As expected, AC, WQ (750 °C and 1000 °C) and FC samples exhibit lower hardness values than WQ (1200 °C), which results in higher strength of the material which is suitable for aerospace applications.

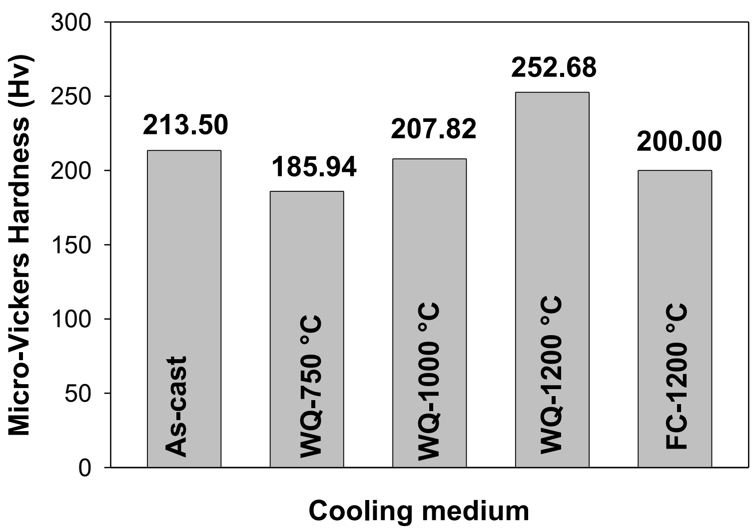


Fig. 4. Micro-Vickers hardness of B2 FeCo alloy at different cooling types: As-cast, WQ at 750 °C, 1000 °C, 1200 °C and FC at 1200 °C.

4 Conclusion

The microstructural and phase analyses as well as hardness tests of the FeCo alloy in the as cast and heat-treated conditions were successfully carried out. The XRD results of as-cast showed the presence of the equiatomic α'-B2 phase which is the stable phase at room temperature. According to XRD analysis, the samples in the AC and FC resulted in one phase: namely α′ with traces of α phase. Those samples exposed to WQ at 750 °C and 1000 °C showed only α phase while sample at WQ (1200 °C) exhibited both α′ and α phases. On OM micrographs of the as-cast FeCo sample, finer equiaxed grains were observed. It was found that the WQ sample (750 °C) had equiaxed grains, but the grains were coarser as compared to the as-cast sample. Compared to all the samples, WQ at 750°C had the lowest hardness value due to the presence of α phase and coarse grains. Additionally, WQ (1200 °C) sample displayed the highest hardness value as compared to both AC and FC samples. Due to different behaviours observed after the alloy is subjected to different cooling media, it can be concluded that different cooling rates influence the resulting microstructure, phases and hardness of FeCo alloy. Understanding the heat treatment of FeCo alloy is essential for achieving desired performance outcomes.

This work was conducted at MINTEK. The authors would like to thank the Advanced Metals Initiative (AMI) of the Department of Science and Innovation (DSI) for their financial support.
Open access funding was provided by MINTEK.
Data availability: Data reported in this paper is available upon request to the corresponding author.

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