Morphology, Structure and Composition of Precipitated Phase in As-Cast Fe35Ni35Cr20Mn10Ti6 High Entropy Alloy

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Abstract: In this study, Fe35Ni35Cr20Mn10Ti6 high entropy alloy was prepared by arc-melting. The microstructure and chemical composition of the alloy were characterized by FIB double beam scanning electron microscope. The result show that there is a typical dendritic structure in the as-cast Fe35Ni35Cr20Mn10Ti6 HEA, which is composed of grey dendritic tissue, white and black island-like inter-dendritic structure. The morphology, crystal structure and composition of the precipitated phases in the as-cast Fe35Ni35Cr20Mn10Ti6 HEA were further determined by transmission electron microscopy. The formation sequence and formation mechanism of various precipitated phases during the cooling process were discussed by microstructure observation and phase calculation.

Keywords: Fe35Ni35Cr20Mn10Ti6 high entropy alloy, Microstructure, Solidification process

1 Introduction

The novel alloy design concept of high entropy alloys has been widely concerned since it was proposed in 2004, and related research work has been vigorously developed [1, 2]. Efforts have been made to improving the comprehensive mechanical properties of FCC HEA, such as fine-grain strengthening, second-phase strengthening, solid-solution strengthening and deformation strengthening, etc., but in the most cases, a significant degree of plasticity is lost [3, 4]. Dual-phase HEA can be strengthened and toughened by using softer matrix phase and harder second phase with sufficient fraction, so it becomes an important point in the research field of HEA.

Liao et al. [5] pointed out that Ti alloying can effectively solve the strength-toughness balance problem of Fe35Ni35Cr20Mn10 HEA. However, it is unclear how the second phase is formed during solidification of dual-phase HEA. Therefore, it is a key scientific problem to elucidate the solidification and crystallization law of dual-phase HEA, especially the formation process of the second phase.

In this study, the as-cast Fe35Ni35Cr20Mn10Ti6 HEA was prepared by arc-melting. The microstructure and chemical composition of HEA was studied, and the formation sequence and formation mechanism of various precipitates during solidification were discussed. It has positive significance for the development of HEA solidification theory.

2 Experimental procedure

The as-cast Fe35Ni35Cr20Mn10Ti6 HEA was prepared by arc-melting. The microstructure and chemical composition of the alloy were characterized by FIB double beam scanning electron microscope and transmission electron microscopy.

3 Result and discussion

Pandat phase diagram

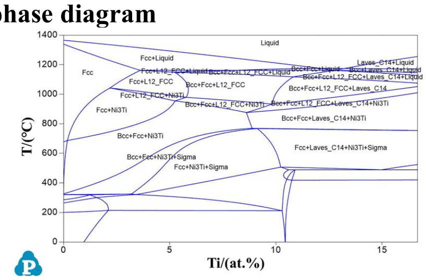

Fig.1 The pseudo-binary phase diagram of (Fe35Ni35Cr20Mn10)-Ti system, calculated by Pandat software

Fig. 1 shows the pseudo-binary phase diagram of (Fe35Ni35Cr20Mn10)-Ti system, calculated by Pandat software. Because of hysteretic diffusion effect of HEA, the phase transformation reactions do not occur at low temperature, and the phase composition above 600°C is shown in Fig.1. It has been shown in literature that Ni and Ti have the largest negative mixing enthalpy and are more likely to aggregate with each other, causing element segregation. This also indicates that the Ti content in each region of Fe35Ni35Cr20Mn10Ti6 alloy is not at a uniform level during the solidification process, so the phase transition in the process is complex. However, it is worth noting that according to the phase diagram, in the range of 3.5%-15% Ti content (Ti content is about 4.8% in the Fe35Ni35Cr20Mn10Ti6), the final solid phases were FCC phase, Ni_3Ti phase, σ phase and Laves phase.

Microstructures

Fig. 2 shows the images of Fe35Ni35Cr20Mn10Ti6 HEA in as cast state (a: low magnification; b: high magnification). The prepared HEA shows a typical dendritic structure, which is composed of grey dendritic trunks, white and black island-like inter-dendritic structure. Spot-scanning results of areas A, B, and C in Fig. 2(b) reveals that the dendritic trunk lacks of Ni and Ti elements compared with the design composition; the inter-dendritic tissue is enriched in Ni and Ti elements; and the content of Ni and Ti in the black island-like structure is further high.

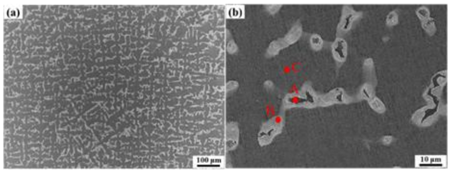

Fig.2 SEM images of as-cast Fe35Ni35Cr20Mn10Ti6 HEA

In order to further analyze the microstructure of the as-cast Fe35Ni35Cr20Mn10Ti6 HEA, the transmission samples of the point C (dendritic trunk) and point A (inter-dendritic zone) in Fig. 2 were prepared by GAI FIB. Fig. 3 shows the TEM bright field image of HEA and the SAED pattern. The dendritic trunk of HEA is a single FCC phase structure as shown in Fig.3(a). As seen in Fig. 3(b), the black island-like structure in Fig. 2 is composed of multiple intermetallic compounds. EDS results of these compounds reveals that the A block structure is rich in Fe, Ni and Ti elements; B block structure is rich in Cr and Fe elements; C block structure is rich in Ni and Ti elements. Combined with the corresponding SADE patterns in Fig. 3, it can be determined that the block structures A, B and C are Laves, σ, and η phases, respectively.

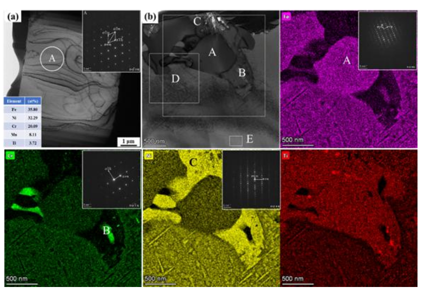

Fig.3 TEM bright-field image and SADE pattern of as-cast Fe35Ni35Cr20Mn10Ti6 HEA：(a) dendritic trunk; (b) inter-dendritic zone

Fig. 4 shows the EDS face-scanning results of areas D and E in the Fig.3 and the SADE patterns of related areas. By the EDS face-scanning results, spherical nanoparticles γ' phase and needle-like η phase can be observed in region D in Fig. 3. Whereas, only spherical nanoparticle γ' phase was seen in region E in Fig. 3. In addition, the Ti content in region C is higher than that in region A in the Fig. 4. This indicates that the level of Ti content has an important effect on the formation of η phase.

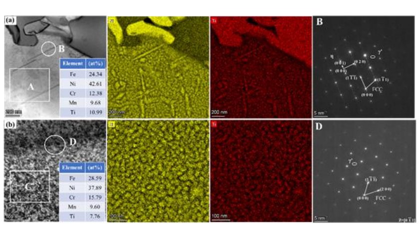

Fig. 4 EDS results and SADE pattern of area D (a) and area E (b) in the Fig. 3

In summary, the as-cast Fe35Ni35Cr20Mn10Ti6 high-entropy alloy has a typical dendritic structure, and the specific structure can be summarized as follows: the phase distribution centered on the Ti rich island-like up to the dendrite region is in the order of Laves + η + σ, FCC + η + γ′, FCC + γ′ and FCC, as shown in Fig.5.

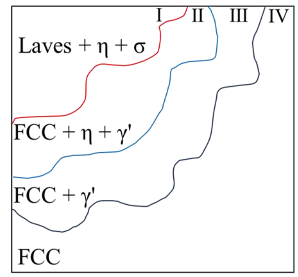

Fig. 5 The microstructure distribution diagram of as-cast Fe35Ni35Cr20Mn10Ti6 HEA.

4 Conclusion

There is a typical dendritic structure in the as-cast Fe35Ni35Cr20Mn10Ti6 HEA, which is composed of grey dendritic trunk, white and black island-like inter-dendritic zone.

The specific structure can be briefly summarized as the phase distribution centered on the Ti rich island-like up to the dendritic region is in the order of Laves + η + σ, FCC + η + γ', FCC + γ' and FCC.

The gradient distribution of Ti elements from dendrites to inter-dendritic zone is the main reason for the gradient distribution of phases

References

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