RESEARCH ARTICLE

Microstructural Refinement for the Reduction of Casting Thickness in Ni-Zr-(Al) Eutectic Composites and Evaluation of the Cooling Rates

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ABSTRACT: The variation of casting thickness and solute content may play a significant role to alter the microstructure, thus, mechanical, creep and other properties. Therefore, the present work aims on the microstructural modification in terms of the length scale of the constituent phases and microstructure homogeneity in differently cast rods (Ø=2 mm, 3mm, and 5 mm) and ingots of $(Ni_{0.92}Zr_{0.08})_{100-x}Al_x$ ($0 \leq x \leq 4$ *at.*%) composites, which are finally correlated with the cooling rates. The composites are comprised of γ-Ni dendrites embedded on a eutectic matrix of γ-Ni and Ni_5Zr lamellar phases. The secondary dendritic arm spacing (SDAS) and average eutectic lamellae thickness (λ_w) are refined from 6.00±0.9µmto 1.75±0.20 µm, and from 275±6 nm to 40±3 nm respectively with the variation of casting thickness. The rods are solidified under very rapid cooling rate upto 1.6×10^4K/s resulting very fine and homogeneous microstructure. The segregation effect is minimised in the smaller diameter rods resulting very uniform microstructure throughout the cast rods.

KEYWORDS: Ultra fine eutectics, casting thickness, Microstructure, Solute content, Cooling rate

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1. INTRODUCTION

The microstructure of any alloys in terms of its homogeneity the evolved phases and grain size change with the variation of solidification time. The velocity of the solid-liquid interface increases with increasing cooling rate, thus, the microstructure gets refined [1-3]. Also, in a large section of cast product, cooling rate may vary to a large extent from the inner region to the outer region which may create non uniformity in microstructure and mechanical properties in the cast alloys [3-5].In case of slow cooling rate, the solute content varies in different portion of the cast alloys. During the equilibrium solidification, the atomic diffusion at solid-liquid interface is much faster than the interface velocity and the composition follows the equilibrium phase diagram [2-6]. As the diffusivity of the solid is significantly lower as compared to the liquid, the solute concentration will be more in the liquid just adjacent to the solid-liquid interface causing the well-known segregation phenomena, which can detriment material's properties. Whereas, the rapid solidification process (RSP) permits the deviation from local equilibrium at the solid-liquid interface [2-6]. As a consequence, solute concentration in solid phase is increased which decreases the segregations in liquid. The solute concentration for the partition-less solidification, is equal in both solid and liquid phases resulting no segregation. Very fine structure with uniform properties can be obtained by this process [3, 6].

Nano-/ultrafine eutectic composites have been developed in several multicomponent Ti-, Fe-, Ni-, Zr- systems with superior mechanical, corrosion and creep resistance properties [7-10]. Recently, low melting temperatureNi-Zr-(Al) ultrafineeutectic alloys, having high strength and large room temperature plasticity, are being developed as an advanced Ni-base alloy [8-10]. These alloys are comprised of γ-Ni dendritic phase homogeneously embedded on a eutectic matrix of γ-Ni and Ni_5Zr lamellae[8, 10].The Al addition increases the volume % of γ-Ni dendrites[9, 10]. However, the rapid solidification in the cast rods may derive many other microstructural features which are yet to be explored. On the other hand, due to very high cooling rate, non-equilibrium composition is expected to evolve in as-cast rods [2,9,10].Therefore, in the present work, the microstructural features have been quantified and are used as parameters to correlate the solidification parameters such as cooling rate, segregation, and homogeneity in the cast rods of diameters 2 mm, 3 mm, and 5 mm and the results are reported.

2. EXPERIMENTAL

The as-solidified ingots (ASI) of the investigated composites of $(Ni_{0.92}Zr_{0.08})_{100-x}Al_x$were prepared by arc melting technology under Ar atmosphere from the pure Ni, Zr, and Al(99.99 wt%). The as-cast rods of the alloys were cast by suction casting technology of the arc melted. The required amount of Al based on the alloy composition, was co-melted with the binary $Ni_{92}Zr_8$ alloy which was prepared preliminarily. Several times remelting were performed in order to mix and homogenise the constituent elements properly before casting. Figure 1 shows few images of the as-cast rods (Ø=2-5

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mm)and ingot of *x*=4 composite. All the rods and ingots were cut by electro-discharge machining (EDM) as per the required size for the microstructural characterisation. Mechanical polishing of the specimens through various stages was performed. Colloidal-silica suspension was used at the final stages of polishing in order to get the mirror-like finished surface. The microstructure of the composites was taken using a optical microscope at a magnification 100 X, 200 X and 500 X to reveal the length scale variation of the micrometer size dendritic phase and the vol.% of the γ-Ni dendritic phases. The field emission scanning electron microscope of FESEM-SUPRA 40, Carl-Zeiss SMT AG (Germany) was used for microstructural characterization to measure the nanometer size lamellae thickness. The secondary dendritic arm spacing (SDAS) and the interlamellar spacing (λ) were estimated for evaluation of solidification parameters [10]. The SDAS has been estimated from the formula SDAS = L/(N-1), where the primary dendrite arm (L) length and the number of the secondary arms (N) in the longitudinal section, was measured at the centre of the rods and ingots. Similarly, interlamellar spacing (λ) was measured taking the parallel set of the lamellae in which λ is the total thickness of the two lamellae ($\lambda_{\gamma\text{-Ni}} + \lambda_{Ni5Zr}$).

3. RESULTS AND DISCUSSIONS

3.1 Microstructure refinement and homogeneity

Figure 2(a-d) shows the optical micrographs of differently solidified *x*=1 alloy at lower magnification. The brighter Ni-rich γ-Ni dendrites, is homogeneously distributed on a darker eutectic matrix in all the differently solidified ingots and rods. It was observed that the vol.% of the γ-Ni dendrites increases with increasing Al content in the alloy. However, there is not much variation of vol.% of dendrite for the similar alloy composition in all ingots and rods. For an example, the vol.%of the dendritic phase varies between 22.8±2.6 % (2mmØ) and 24.4±2.5 % (5mmØ) for the *x*=1 alloy. It is observed that the secondary dendritic arm spacing (SDAS) reduces gradually with decreasing casting thickness. The SDAS is refinedfrom6.00±0.9 µm (ASI) to 1.75±0.20 µm (2 mmØ) with reducing casting thickness. The higher magnification SEM back-scattered electron (BSE) micrographs of *x*=0 alloys is shown in Figure 3(a-d). There is large reduction of interlamellar spacing in smaller diameter rods. The eutectic interlamellar spacing (λ) is refined from 550±12 nm (ASI) to 78±6 nm (2 mmØ) respectively with the variation of casting thickness [10]. Al dissolved predominantly in the γ-Ni phase as substitutional solid solution [9, 10]. Whereas in the rapidly solidified 3 mmØ rods, the small variation of the interlamellar spacing along the cross section i.e. from centre to the periphery is observed. The variation of average lamellae thickness ($\lambda_w = \lambda/2$) is only between 51±5 nm and 63±4 nm; and between 52±5 nm and 60±4 nm for *x*=0 and *x*=4 composites respectively [10]. Thus, it can be said that the microstructure remains homogeneous and uniform throughout the section in the rapidly cooled 3 mmØ cast rods. Also, as the SDAS is reduced with reducing casting thickness and less segregation is occurred for smaller diameter rods.

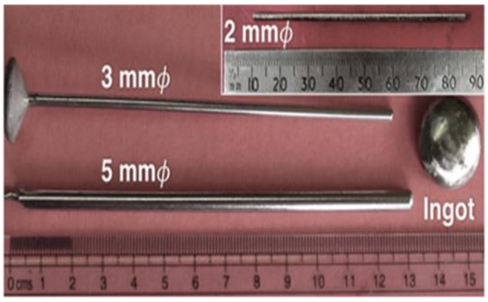

Figure 1. The outer appearance of rods of varying diameters and ingot

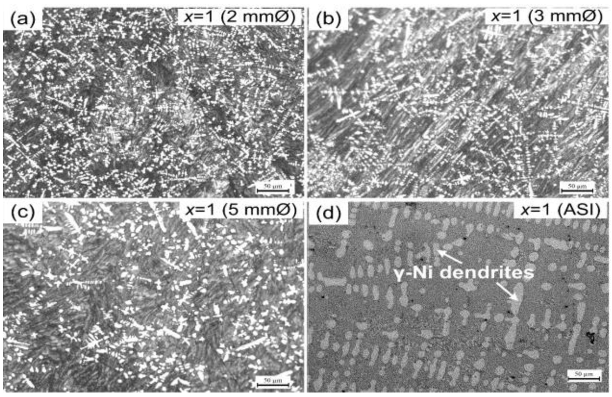

Figure 2. Optical micrographs of *x*=1 alloys of differently solidified ingots and rods at lower magnification showing the variation SDAS with casting thickness.

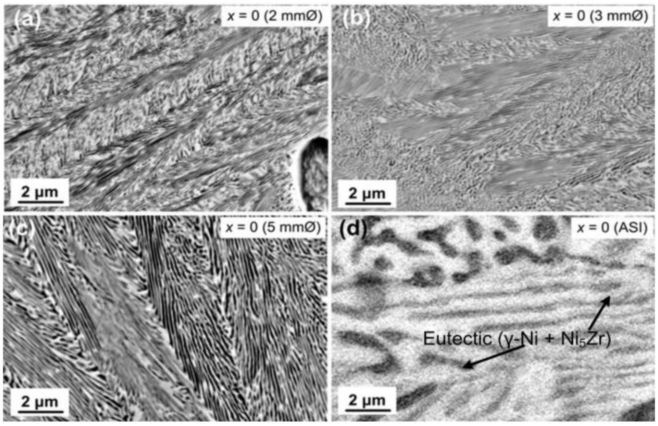

Figure 3. SEM BSE micrographs of *x*=0 composites at higher magnification showing the refinement of lamellae thickness with casting thickness.

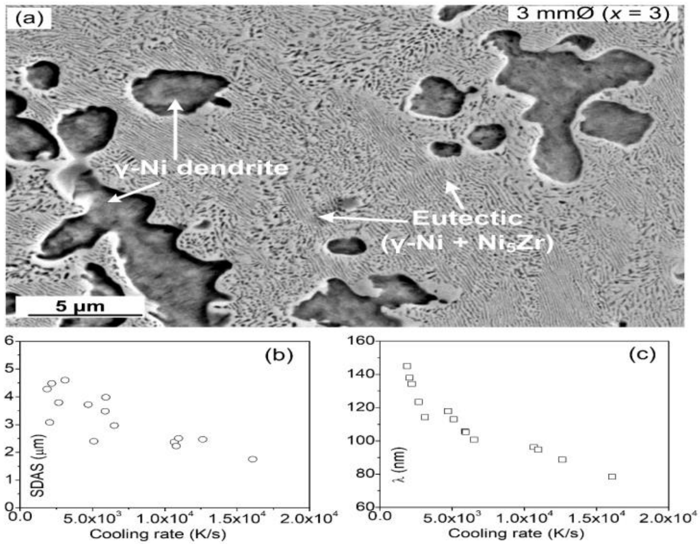

Figure 4. (a) SEM BSE micrograph of the 3 mmØ rod of *x*=3 alloy, the variation of (b) SDAS (µm) (c) *λ* (nm) with cooling rate (K/s)

3.2 Microstructural refinement with cooling rate

The cooling rates of the differently solidified as-cast rods were calculated as per the Jackson-Hunt model from the measured interlamellar spacing as follows [11]:

$$v \times \lambda^2 = K \quad .... \quad (1)$$

The mold is cylindrical in which the solidification takes place from the outer surface. The cooling rate ($\dot{T}$) is derived from the following equation from the solidification velocity (v) in which R, Δh_f, and C_p, are the radius of the rods, heat of fusion and the specific heat capacity respectively [12]:

$$\dot{T} = \frac{\Delta h_f}{C_p} \times \frac{2v}{R} \quad .... \quad (2)$$

Therefore, by using the above two equations cooling rate is calculated from the measured eutectic interlamellar spacing (λ) as follows [12]:

$$\dot{T} = \frac{\Delta h_f}{C_p} \times \frac{2\boldsymbol{K}}{R\lambda^2} \quad .... \quad (3)$$

In the investigated Ni-Zr-(Al) system, the solidification constant (K)is taken as 100×10^{-12} cm^3/s [13-17]. The value of $\Delta h_f/C_p$ for the Ni-Zr-(Al) system is calculated to be 485 K [13-17].

Figure 4(a) show the the SEM BSE micrographs of the 3 mmØ rod in which γ-Ni dendrites are homogeneously embedded on a eutectic matrix of γ-Ni and Ni_5Zr lamellae. The measured SDAS for this study along with the reported values of SDAS and interlamellar spacing (λ) is plotted with the cooling rate as calculated from λ by Jackson-Hunt model in Figs. 4(b-c)[10]. It is clearly observed that the SDAS and λ decreases with decreasing casting thickness. Whereas, there is large alteration of the cooling rate due to change in casting thickness Figure 4(b-c).

4. CONCLUSION

The present study concludes that the microstructure of all the differently solidified rods and ingots are comprised of micrometer size γ-Ni dendrites on nano/ultrafine eutectic lamellae of γ-Ni and Ni_5Zr phases. However, the SDAS and λ_w are refined with increasing cooling rate and reducing solidification time for the reduction of casting thickness. All the rods were solidified very rapidly in which cooling rate reaches up to 1.6×10^4 K/s for 2 mm Ø rods, resulting very fine, homogeneous and uniform microstructure. The segregation of solute becomes less due to the rapid solidification with the refinement of microstructure in terms of SDAS and λ.

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