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Microstructure and properties of Ni–Ni_3Si composites by directional solidification

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ABSTRACT

Ni–Ni_3Si composites are prepared by the Bridgman directional solidification technology under different growth conditions, aiming to improve the ductility of the Ni_3Si compound and investigate the relationship between solidification microstructure and the properties. Microstructure of the Ni–Ni_3Si hypoeutectic *in situ* composites transforms from regular lamellar eutectic to cellular structure then to dendritic crystal with the increase of the solidification rate. Ni–Ni_3Si eutectic composites display regular lamellar eutectic structure at the solidification rate R=6.0–40.0 μm/s and the lamellar spacing is decreased with the increase of the solidification rate. Moreover, the Ni–Ni_3Si hypoeutectic composites present lower micro-hardness than pure Ni_3Si, which indicate Ni–Ni_3Si hypoeutectic composites have higher ductility, whereas the ductility of the Ni–Ni_3Si eutectic composites has scarcely been improved. This is caused by the formation of the metastable $Ni_{31}Si_{12}$ phase in the Ni–Ni_3Si eutectic composites.

1. Introduction

Due to high strength, low density and elevated thermal stability, intermetallic compound materials have the greatest potential to meet ever increasing requirements of the automobile, aeronautic and aerospace industry. Among those materials, the Ni_3Si compound has been paid more attention, because it has many characters, e.g. high melting point, high strength, low density, excellent oxidation resistance at elevated temperatures, and magnificent corrosion resistance in acid environments, particularly in sulfuric acid solutions [1–6]. However, the engineering application of the Ni_3Si compound has been limited by its poor ductility at ambient temperatures and its bad fabricability at high temperatures [6,7]. The grain boundaries in Ni_3Si compound are also intrinsically brittle, like in the majority of ordered $L1_2$ intermetallics, resulting in a brittle intergranular fracture. Much work has been done to improve the ductility of the Ni_3Si compound, for example, disordering treatment, alloying, microstructure control, and composition, etc.

The incorporation of a ductile phase into the intermetallic materials is an attractive means to improve the ductility of the intermetallic materials. This can be achieved by directional solidification processing of eutectic alloys, and eutectic *in situ* composites which are thermodynamically stable, chemically compatible, and well aligned. Dyck et al. [8] reported Ni–Ni_3Si eutectic *in situ* composite by powder metallurgy technique. Dutra et al. [6] and Milenkovic and Caram [9] obtained Ni–Ni_3Si eutectic *in situ* composite with the Bridgman directional solidification technique at solidification rates R=32–56 μm/s. Hui et al. [10] adopted the micro-denucleation technique of bulk melt to obtain 224 K undercooling of the Ni–Ni_3Si composite and studied the growth mechanisms of the Ni_3Si phase. Although many significant results have been achieved, the crystal growth mechanism and the relationship between the solidification microstructure and the properties have not been thoroughly studied. There are still a series of unsolved problems such as the microstructure control, the N–Ni_3Si interface structure and the phase composition of the Ni–Ni_3Si composites. This paper reports the preparation of the Ni–Ni_3Si composites by the Bridgman directional solidification technique at sub-high rates. The eutectic microstructure is obtained at a wide composition range by increasing solidification rate and temperature gradient in front of the solid–liquid interface. Microstructures of the directionally solidified Ni–Ni_3Si eutectic and Ni–Ni_3Si hypoeutectic are studied in detail. In general, the harder the metal material is, the worse its ductility is. Therefore, micro-hardness can be used to represent the ductility of the Ni–Si alloy. Micro-hardness of the Ni–Ni_3Si eutectic and Ni–Ni_3Si hypoeutectic composites at the different solidification rates are studied by micro-hardness tester. Finally, the relationship among solidification rate, alloying constituent and micro-hardness is obtained.

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2. Experiments

The master alloys are obtained by cutting the middle of the Ni–9 wt% Si hypoeutectic alloy and the Ni–11.5 wt% Si alloy into $\Phi 6 \times 30$ mm slices, which are produced with the vacuum arc melting technique. The Ni–Ni_3Si hypoeutectic (eutectic) *in situ* composites are prepared by the Bridgman directional solidification technique at sub-high rates. The directionally solidified samples are treated with a conventional metallographic technique and etched by the mixture of 5%HCl+H_2O+Fe_3Cl solution, and the microstructure and phase distributions are observed with OLYMPUS GX51 optical microscope. Micro-hardness of the Ni–Ni_3Si hypoeutectic (eutectic) composites at different solidification rates are studied by the 40MVD microhardness tester. Phase composition of the Ni–Ni_3Si hypoeutectic (eutectic) *in situ* composites are studied by the X-ray diffraction (X Pert MPDPRO) technique.

3. Results and discussions

3.1. Microstructure and micro-hardness of the Ni–Ni_3Si hypoeutectic *in situ* composites

Microstructure of the Ni–Ni_3Si hypoeutectic *in situ* composites on the longitudinal direction is shown in Fig. 1. It clearly shows the composite microstructure of Ni_3Si compound (light phase) in Ni matrix (dark phase). It can be seen from Fig. 1 that the solidification microstructure is a regular lamellar eutectic structure at the solidification rate R=3.0–8.0 μm/s as shown in Fig. 1a and b. The directional heat flow perpendicular to the solid–liquid interface and a high temperature gradient in front of the solid–liquid interface are the two important factors to guaranty the directional growth of crystals. In the present study, the directional heat flow and higher temperature gradient can be achieved by using Ga–In–Sn alloy as coolant and the thermal shield to separate the cool and the hot zones. Meanwhile, the positive temperature gradient maintains the planar solid–liquid interface. Therefore, the lamellar eutectic structure is obtained at the lower solidification rate as shown in Fig. 1a and b. In general, a higher solidification rate can lead to a bigger supercooling. With the increase of supercooling, the nucleation rate and growth rate of Ni_3Si phase are increased, whereas the diffusion rate of solute in the liquid is decreased. If the solidification rate is relatively small, the nucleation rate will play the main role and result in the refinement of the solidification microstructure, which is why the lamellar spacing of Ni–Ni_3Si hypoeutectic *in situ* composite is decreased with the increase of the solidification rate as shown in Fig. 1a and b. Constitutional supercooling at the solid/liquid interface is gradually increased with the increase of the solidification rate. When the constitutional supercooling is large enough to destroy the planar solid–liquid interface, the cellular crystal appears when the solidification rate reaches 25 μm/s as shown in Fig. 1c. With the further increase of the solidification rate, the solid–liquid interface becomes more unstable, the deep cellular structure appears, and those small cellulars then start to grow and eventually transform into dendrites as shown in Fig. 1d. Moreover, the lamellar spacing of Ni–Ni_3Si hypoeutectic *in situ* composite is synchronously decreased with the increase of the solidification.

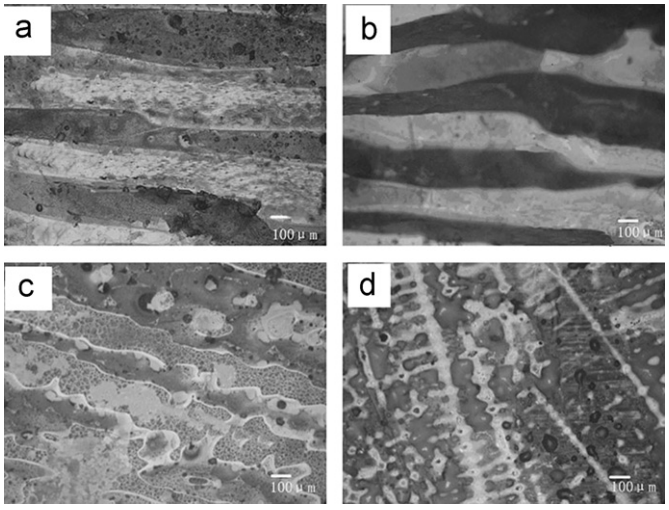

Fig. 1. Longitudinal microstructure of the Ni–Ni_3Si hypoeutectic *in situ* composites at different solidification rates. (a) R=3 μm/s, (b) R=8 μm/s, (c) R=25 μm/s and (d) R=40 μm/s.

Indentation pattern of the Ni–Ni_3Si hypoeutectic *in situ* composite is shown in Fig. 2. Table 1 shows the micro-hardness of the Ni–Ni_3Si hypoeutectic *in situ* composites at different solidification rates. The relationship between solidification rate and the micro-hardness of the Ni–Ni_3Si hypoeutectic *in situ* composites is shown in Fig. 3, which demonstrates that the micro-hardness of the Ni–Ni_3Si hypoeutectic *in situ* composites is first decreased and then increased with the increase of the solidification rate. This is caused by the change of microstructure of the Ni–Ni_3Si hypoeutectic *in situ* composite. The optimal microstructure of the Ni–Ni_3Si hypoeutectic *in situ* composite is obtained when the solidification rate is R=8.0 μm/s and the microhardness of that is the minimum as a result.

In comparison with the micro-hardness of pure Ni_3Si compound [11], micro-hardness of the Ni–Ni_3Si hypoeutectic *in situ* composite is greatly decreased. That is to say, the ductility of the Ni–Ni_3Si hypoeutectic *in situ* compound is greatly improved when Ni_3Si compound is combined with the Ni matrix.

3.2. Microstructure and micro-hardness of the Ni–Ni_3Si eutectic *in situ* composites

Microstructure of the Ni–Ni_3Si eutectic *in situ* composites on the longitudinal direction is shown in Fig. 4. It clearly shows the

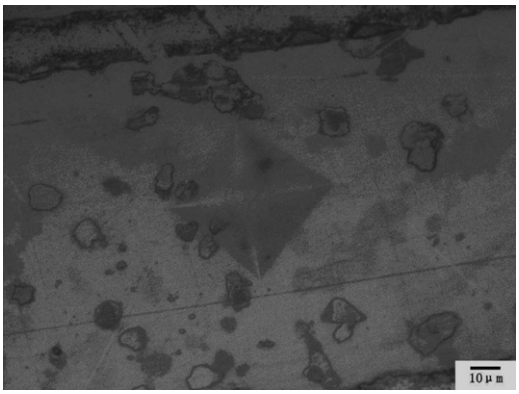

Fig. 2. Indentation pattern of the Ni–Ni_3Si hypoeutectic *in situ* composite.

Table 1
Micro-hardness of the Ni–Ni_3Si hypoeutectic *in situ* composite at different solidification rates.

Sample	Micro-hardness (Mpa)
Ni–Ni_3Si hypoeutectic *in situ* composite (R=3.0 μm/s)	267.5
Ni–Ni_3Si hypoeutectic *in situ* composite (R=8.0 μm/s)	226.8
Ni–Ni_3Si hypoeutectic *in situ* composite (R=25.0 μm/s)	262.8
Ni–Ni_3Si hypoeutectic *in situ* composite (R=40.0 μm/s)	285.1
Pure Ni_3Si compound	716