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High-temperature tensile properties of a NiTi–Al-based alloy prepared by directional solidification and homogenizing treatment

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ABSTRACT

To develop a new type of light-weight, high strength, high-temperature structural material, cast bars of a NiTi–Al-based alloy with low Nb and Hf contents were prepared by DS (directional solidification). The cast bars prepared by optimal DS were homogenizing treatment at 950 °C for different times. The tensile strength of the homogenized test samples at elevated temperatures was tested. The results reveal that the DS microstructure is a cellular-dendrite or acicular cellular structure that preferentially grows along the [001] orientation. With an increase in the drawing velocity, the cellular crystals and cellular arm spacing gradually decrease, but the directionality of the structure weakens. With an increase in the homogenization time, the cellular structure becomes increasingly coarse, and the precipitated phases are more dispersed and distributed more homogeneously. At 800 °C and 850 °C, the highest tensile strengths occur with 12 h of homogenizing treatment, resulting in strengths of up to 340 MPa and 263.6 MPa, respectively; at 900 °C, the tensile strength is 171.5 MPa. This alloy is expected to become a high-temperature structural material for applications at above 800 °C. The highest elongation at 800 °C is 42.4%, and at 850 °C and 900 °C, the highest elongation is 47.2% and 70.8%, respectively. The alloy exhibits good plasticity at high temperatures.

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1. Introduction

To meet the demand of aeronautic and astronautic devices for new-style lightweight high-temperature structural materials, scientists have focused on the lightweight and high-strength intermetallics. However, most intermetallics have not been widely used because of their poor room-temperature ductility [1–4]. γ -TiAl based intermetallics have been attracted great attention by scientists for decades for their low density and high-temperature high strength. However, besides the poor room temperature ductility, γ -TiAl based alloys can only be used for lowpressure blades, and generally, the service temperature is lower than 800 °C [5]. Therefore, seeking a low density, good room temperature ductility and high-temperature high strength of structural material to substitute the large specific weight of nickel base superalloy has great significant.

NiTi-based intermetallics are excellent alloys and are favored by the materials field for their low density (6.2 g/cm^3), excellent shape memory, superelasticity and high room temperature ductility [6-10]. Since Koizumi et al. [11] added different amounts of Al to NiTi alloys as a substitute for Ti in nearly equal atomic ratios and found that the compression strength improved dramatically at room temperature and at high temperature, NiTi-Al-based alloy is considered as a potential high temperature structural material. In recent years, research has focused on adding alloying elements to NiTi-Al alloys in an attempt to further improve their high-temperature mechanical properties. Meng et al. [12,13] found that Nb can improve the compression strength of a NiTi-Al alloy from room temperature to 700 °C and that the compression yield strength and specific strength of the $\rm Ti_{50}\rm Ni_{40}\rm Al_8\rm Nb_2$ alloy are as high as 1237 MPa and 216 MPa g^{-1} cm³, respectively, at 600 °C, exceeding the properties of the René 95 superalloy. In addition, Zhao et al. [14] reported that Nb can improve the oxidation resistance of NiTi-Al-based alloys at approximately 800 °C. Song et al. [15] researched the strengthening effect of elemental Mo on NiTi-Al-based alloys and found that the highest compression yield strength at 800 °C is associated with the Ni₅₀Ti₄₃Al₆Mo₁ alloy. At present, NiTi-Al-based alloys have been deemed a potential hightemperature aerospace structural material for applications on top of the mesothermal interval (800-900 °C).

In addition, basic researches of using new preparation technic (such as DS (directional solidification)) to prepare NiTi-Al-based alloy are reported. Pan et al. [16] studied the effect of casting temperature on microstructure in a directionally solidified Ni-44Ti-5Al-2Nb-1Mo alloy, which found that the [100] orientation is significantly enhanced and the solid/liquid (S/L) interface





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morphology changes from cellular dendrite to cellular with the casting temperature increase. Sang et al. [17] investigated the directionally solidified Ni-43Ti-4Al-2Nb-2Hf alloy at different casting temperatures, which found that the increases of casting temperature make the dendrites finer and the precipitate phase more dispersively distributed. According to Sang's report [18], the room temperature tensile strength of the directionally solidified Ni-43Ti-4Al-2Nb-2Hf alloy is as high as 1739 MPa, and the elastic strain surpasses 3%. This ductility can rarely be obtained for many hard brittle intermetallics. However, as far as we know, the high-temperature mechanical properties of NiTi-Al-based alloys that were prepared by directional solidification have not been reported. In this paper, based on research works of Sang et al. [17], the microstructure and high-temperature tensile properties of the Ni-43Ti-4Al-2Nb-2Hf allow that was prepared by DS at various drawing velocities and for different homogenizing annealing times are reported: the results provide a reference for the development of a new type of high-temperature structural material.

2. Experimental procedures

High purity Ni (99.95%), Ti (99.99%), Al (99.99%), Nb (99.98%) and Hf (99.9%) were used as the raw materials to prepare master alloy ingots with nominal compositions of Ni-43Ti-4Al-2Nb-2Hf (at.%). The master alloy ingots were prepared by arc-melting on a water-cooled copper crucible in an argon atmosphere and were melted four times to ensure composition homogeneity. Before the DS experiment, the bar stock for DS was cut by wire-electrode cutting to 14 mm in diameter and 220 mm in total length from the master alloy ingots. The surface oxide skin of the bar stock was lathed off, and the bar stock was ultrasonically washed in acetone solvent and then dried. Finally, the bar stock was packed into a Y_2O_3/Al_2O_3 double-layer ceramic tube (\emptyset 14.5 × 240 mm) similar to that reported by Zhang et al. [19], which acted as the melting crucible, and then placed in a laboratory-scale Bridgman-type directional solidification furnace (LMC, liquid metal cooling technology). Before each experiment, the furnace chamber was vacuumized to $3-5 \times 10^{-3}$ Pa and then backfilled with high purity argon to a pressure of 0.05 MPa. The holding temperature and holding time were 1550 °C and 20 min, respectively. After reaching the holding time, the ceramic tube and the melt inside were drawn into the Ga-In-Sn cooling liquid at different drawing velocities (2 mm/min, 18 mm/min, 30 mm/min and 60 mm/min). In order to eliminate the nonuniformity of the intercrystalline chemical composition and structure, the DS bars were vacuum homogenizing annealing treated at 950 °C for 12 h, 50 h and 100 h, (furnace cooling), and they were then processed into tensile specimens with the dimensions \emptyset 5 × 35 mm (parallel length) (shown in Fig. 1).



Fig. 1. Photo of tensile specimen.

The microstructural samples were sampled from the centers of the DS bar stock, which are the steady-state growth regions. The samples were cut in longitudinal sections and transverse sections and then ground and polished. The microstructure and composition of the samples were analyzed with a JXA8100 electro-probe microanalyzer (EPMA) (JEOL, Japan), which was equipped with energy dispersive X-ray spectroscopy (EDS). The phase types were analyzed with a D/max2200pcX-type X-ray diffractometer (XRD) with Cu K α radiation (voltage: 40 kV; current: 40 mA; scanning speed: 6°/min; scanning range: 20–90°). The Cambridge 3400 scanning electron microscope (SEM) was used to observe the fracture surface morphology. The IAS8 metallographical analysis software was used to the phase volume statistical analysis. The high temperature (800 °C. 850 °C and 900 °C) tensile tests were performed with a Shimadzu IS-10T electronic testing machine in atmospheric conditions. The holding time before stretching was 20 min, and the tensile rate was 0.5 mm/min. The elongations were measured with an electronic digital caliper with a precision of 0.01 mm.

3. Results and discussion

3.1. XRD analysis of the samples

The XRD diffraction spectra of the samples are shown in Fig. 2. It can be observed that the master alloy sample and the DS samples are composed of the NiTi, β -Nb and Ti₂Ni phases. The phase types do not change after DS. The phase types are the same as the $Ni_{45.5-}$ Ti_{45.5}Al₆Nb₃ alloy that was investigated by Jian Xu et al. [20], the Ni₅₀Ti₄₈Nb₂ and Ni_{49.5}Ti_{46.5}Nb₄ alloys that were reported by Xiao Fu et al. [21], and the $Ni_{50.1}Ti_{46.9}Nb_3$ alloy that was researched by He et al. [22]. With the increase in the drawing velocity, the NiTi phase is inclined to preferentially grow along the (100) and (200) crystallographic planes, while several orientation peaks appear in the as-cast of master alloy sample. The diffraction peaks of the β -Nb phase and the Ti₂Ni phase are very weak. In some samples, their diffraction peaks cannot be found at all, and their crystal orientations demonstrate no obvious changes with the increase in the drawing velocity. We assume that these results are related to the low volume content of the β -Nb phase and the Ti₂Ni phase in the alloy. Based on the volume fraction statistics shown in Table 1, the volume fraction of the β -Nb phase is approximately 3–6%, and the Ti₂Ni phase is less than 1%.

3.2. Microstructure of master alloy ingot

An EPMA photograph of the master alloy ingot (as-cast) microstructure is shown in Fig. 3, in which it can be seen that the



Fig. 2. XRD diffraction curves of master alloy sample and DS samples.

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