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Comparative study on microstructure and homogeneity of NiTi shape memory alloy produced by copper boat induction melting and conventional vacuum arc melting

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ABSTRACT

A comparative study was carried out on the microstructures of NiTi shape memory alloy produced by two techniques of copper boat induction melting and vacuum arc melting. The as-solidified and homogenized specimens were characterized by optical microscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) equipped with EDS, and differential scanning calorimetry (DSC). The results showed that the microstructure formed in the copper boat method was free of segregation (Ti or Ni-rich precipitates) with a low scatter (<5 K) in phase transition temperatures within the ingot at different positions. Unlike the multi-stage vacuum arc remelting process, a homogeneous microstructure was achieved only after one melting procedure.

1. Introduction

NiTi Shape memory alloy (SMA) has many applications in industry and medicine because of its unique properties such as pseudoelasticity, thermal memory, corrosion resistance and biocompatibility [1–3]. Synthesis of NiTi alloy has always been a challenge for scientists. Phase transition temperatures (PTTs) and functional properties of this alloy are strongly affected by chemical composition and process parameters. In recent years, ingot metallurgy processing of NiTi shape memory alloy has been discussed in great details [4–7]. It is well known that in vacuum induction melting (VIM) method, good chemical homogeneity is achieved by electromagnetic stirring. On the other hand, Ti reacts with graphite crucibles and TiC particles are formed. Formation of these particles changes the alloy composition and as a result, alters the transformation temperatures [4,5,8–10]. In vacuum arc melting (VAM), there is another drawback. Although the problems associated with graphite crucibles are eliminated using the copper molds, due to the lack of enough stirring, chemical homogeneity of the ingots requires several remelting procedures. During these remelting steps, the alloy picks up oxygen and carbon and again the PTTs are affected [6].

In view of the above-mentioned drawbacks in VIM and VAM methods, a new process is needed to cast high quality ingots and to impede any scatter in experimental data for the future research of NiTi SMAs. In this study, small NiTi ingots (weighing about 25 g) were produced by a new process called copper boat induction melting (CBIM). In CBIM, similar to VIM, electromagnetic stirring results in excellent chemical homogeneity but graphite crucible is replaced with water-cooled copper mold (copper boat). Hence, the reaction between Ti and C is eliminated and a clean melt is achieved. Considering microstructures and characteristic temperatures, we will show that better quality is achieved for NiTi SMA.

2. Materials and experimental procedures

Fig. 1 depicts real pictures of CBIM and VAM methods for synthesis of NiTi alloy containing 50.3 at.% Ni. Commercially pure Ni and Ti were put inside an indentation (boat) on a copper tube along with a Ti getter. Then, the copper tube is centered inside a quartz tube to seal it from the atmosphere. Finally, the induction coil is placed around the quartz tube. Copper boat melting was carried out in a sealed pure Ar (99.9998 Vol.%) atmosphere using a radio frequency (RF) induction generator with the working frequency of 330 kHz and nominal power of 45 kW. Prior to melting the raw materials, a Ti getter was melted and held in liquid state for 3 min to get the residual oxygen and nitrogen and to purify the tube atmosphere. Strong electromagnetic stirring provided by induction

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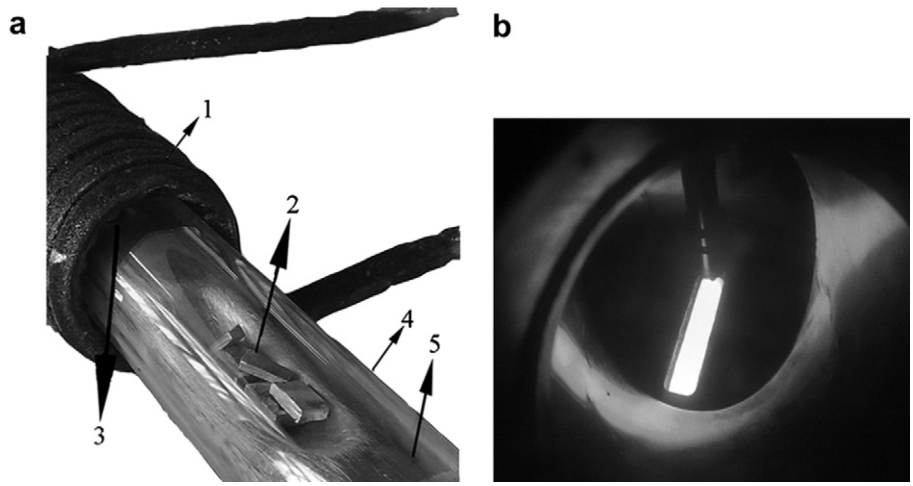

Fig. 1. The real pictures of the (a) CBIM and (b) VAM set-ups. (1) Induction coil, (2) Ti and Ni pieces, (3) Ti getter in a nearby indentation, (4) quartz tube, and (5) high pressure water-cooled copper tube.

coil and lack of reaction between the melt and the copper boat makes the CBIM superior compared to VAM and VIM methods. Arc melting was carried out in pure Ar (99.9998 Vol.%) at a pressure of 700 mbar using a non-consumable vacuum arc remelting furnace made by ALD Germany. Prior to melting, the bell jar was twice evacuated to a pressure of 5×10^{-3} mbar and filled back with argon. To promote thorough mixing, the bar was turned over after each arc melting step, and this procedure was then repeated 4 times. More details of VAM technique can be found elsewhere [6]. The CBIM specimen was homogenized at 1230 K for 2 h whereas the VAM specimen was homogenized at 1273 K for 12 h. After homogenization, both specimens were quenched in water.

Specimens were analyzed by a variety of characterization techniques including optical microscopy with polarized light, SEM (Philips XL30 and Zeiss MA15) equipped with EDS, XRD (Philips X'Pert with Cu Kα anode) and differential scanning calorimetry (Mettler-822e DSC instrument) with the heating/cooling rate of 3 K min^{-1}. Metallographic specimens were mechanically ground on SiC papers (up to 2400 mesh size), polished with abrasive Al_2O_3 powder and etched with a solution composed of 5 ml Hf, 15 ml HNO3 and 50 ml double distilled water.

3. Results and discussion

Fig. 2 shows the optical and SEM micrographs of as-solidified NiTi alloy produced by CBIM and VAM methods. As can be seen, the cast ingots in either method are characterized by large columnar grains with several millimeters in size. The solidification in both methods starts from the bottom of ingots, where the melt is in contact with water-cooled copper. The EDS analysis revealed that Ti- and Ni-rich precipitates (Ti_2NiO_x and Ni_3Ti) were present in the microstructure of VAM, whereas in ingots obtained by CBIM

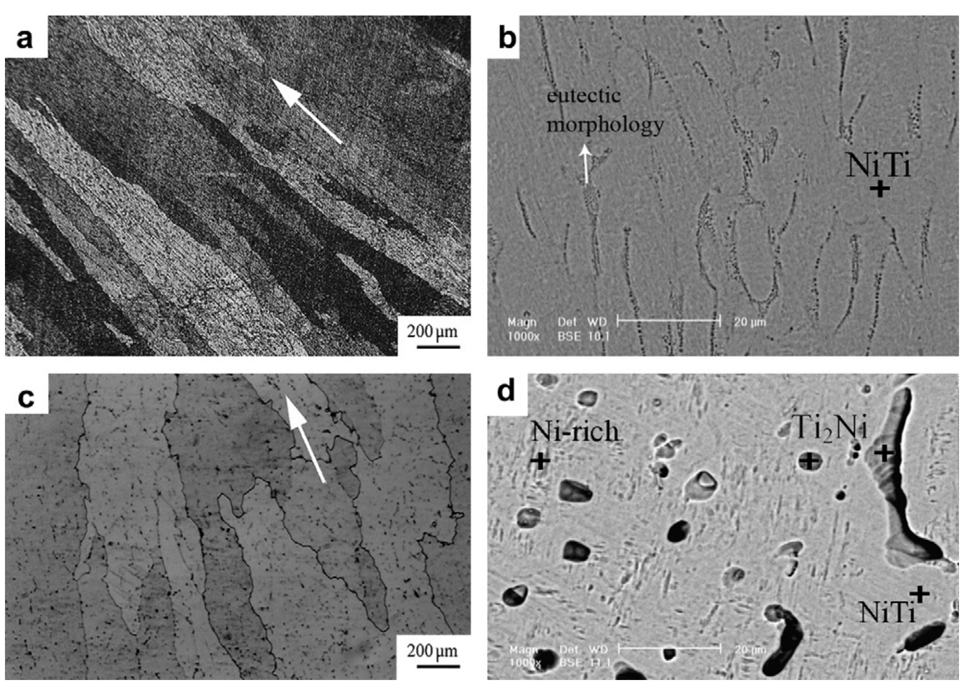

Fig. 2. The optical and SEM micrographs of the as-solidified NiTi ingots produced by (a, b) CBIM and (c, d) VAM methods. The white arrows indicate the solidification direction.