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PROCESSING AND CHARACTERIZATION OF Ni-Al-Fe-B SHAPE-MEMORY ALLOY WIRES PRODUCED BY RAPID SOLIDIFICATION

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ABSTRACT

This work describes net-shape ductile wires of Ni-Al-Fe doped with boron produced directly from the melt by in-rotating-liquid (IRL) melt spinning, thus avoiding the difficult and costly problem of fabricating bulk castings. This method produces wires of 0.1 to 0.5 mm diam and lengths to 2 m. X-ray diffraction (XRD) scans showed that the as-spun wires consist of the B2, L1₂, and bct martensite phases and that the B2 phase further transforms to bct martensite upon cold working. Shape-memory behavior showed an A_p temperature of ~180°C as measured by bend recovery tests and by tensile cycling tests. The effects on the wires of IRL processing parameters are discussed.

1. INTRODUCTION

A number of metallic wires have been cast directly from the melt by the IRL technique [1-3]. The advantage of this method was obvious in that the production of net-shape wire avoids the difficult and costly problem of fabricating bulk castings. Rapid solidification of the wires may produce amorphous or fine-grained crystalline structures and super-saturated alloy phases with compositions that are not obtainable by conventional ingot casting.

Alloys of NiAl with nickel compositions of 61 to 68% are candidate materials for high-temperature shape-memory applications due to their high martensite stability [4-11]. These alloys can be fabricated at elevated temperatures but were brittle with poor ductility at room temperature [4, 12-16]. Although these problems have largely been overcome by the addition of iron and boron [4, 17-22], the mechanical production of a usable form of the alloys is time, labor, and cost intensive.

The alloy wires produced in this study (M_s ~180°C) showed shape-memory behavior measured by bend-recovery tests and tensile cycling tests. The shape-memory effect was quantified by tensile measurements and the alloy phases identified by XRD.

2. EXPERIMENTAL PROCEDURES

Although several alloys with different chemical

compositions were studied, this paper will consider only one, chosen due to its high A_p temperature, ~180°C. This alloy, designated SMA-48, consists of the following atomic percentages: 24.8% Al, 17.1% Fe, 0.2% B, and balance Ni. Assuming that the iron occupies the nickel and aluminum sites equally, the equivalent aluminum concentration [4] is 33.5%. The bulk alloy was arc melted, drop cast into 2.5 x 1.25 x 12.6 cm ingots, examined for macro-segregation, and sectioned into small pieces for melt spinning.

Processing included melting in a quartz crucible by induction heating, then ejecting a liquid stream by an overpressure of argon gas through a small orifice into a layer of rotating liquid. The entire heating, melting, and ejection cycle occurred within 1 min to lessen any reaction between the melt and crucible. A conventional melt-spinning apparatus was altered to accept a cupped aluminum wheel for holding a liquid coolant. The coolant was held on the interior surface of the wheel by centrifugal force. The wheel velocity, ejection pressure (jet velocity), incident angle of the crucible, nozzle diameter, nozzle-to-coolant distance, temperature, and the depth of the coolant layer were variable parameters that affect the wire morphology. The crucible, ejection piping, and induction heater coil were located as far to the rear of the wheel as possible to achieve a small incident angle between crucible and coolant. The crucible was only ~10 cm long and 1.0 cm diam due to the constraints of the wheel size (30 cm).

The wires were examined using both optical and scanning electron microscopy (SEM). Room-temperature XRD of both as-spun and stressed samples was made using Cu K α radiation.

The shape-memory effect was characterized by bend recovery and tensile strain recovery tests. The bend test consisted of bending the sample around a ~3 mm mandrel to produce a 40° angle. The sample was then heated to 300°C, and the bend angle recovery was measured as a function of temperature. A more sensitive tensile strain recovery was measured with the wire sample loaded in a tensile test load frame.

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3. RESULTS AND DISCUSSION

3.1 IRL Melt Spinning

The development of present-day IRL melt spinning has evolved from a history of looking for alternative methods for the fabrication of wires and fibers. The low viscosity and high surface tension of metals have made methods involving a molten jet difficult due to the lack of stability of the liquid stream. The jet may be stabilized by the formation of an oxide "skin" on the surface of the melt stream that supports the liquid until it solidifies within the coolant layer [23 -25]. Argon was used as an over-gas and for ejection, but the experiment was conducted in air to allow the oxygen and nitrogen to react with the aluminum in the alloy and provide a stabilizing layer on the jet. The crucible-to-coolant distance was necessarily long (~2.5 cm) to allow time for the formation of a surface layer, although from a purely melt spinning consideration, the distance should be short.

A photograph of the IRL system during spinning is shown in Fig. 1. The spun wire can be seen as the dark line within the liquid coolant layer. Shape-memory alloy wires of 0.1 to 0.5 mm diam and lengths to 2 m were produced by this method. Ductility of the samples was demonstrated by winding wires into springs around mandrels ≥ 3 mm diam. Wires with diameters of ≤ 0.25 mm had fewer defects than larger ones.

Fig. 1. Photograph of IRL melt spinning apparatus.

3.2 Microstructure

Typical cross sections of as-spun wires examined using optical and SEM metallography

revealed columnar surface grains and dendritic structures in the interior portion. A longitudinal section of an as-spun wire is seen in Fig. 2. At higher magnification, the microstructure, Fig. 3, consists of light-colored near-surface columnar and dendritic grains of the metastable NiAl (B2) phase together with a second phase of Ni₃Al (L1₂). The martensitic bct phase found by XRD was not resolved optically in the as-spun wire.

Fig. 2. Longitudinal view of as-spun SMA-48 wire showing columnar surface grains. Optical micrograph, 200X.

Fig. 3. Optical micrograph of near-surface region of an as-spun SMA-48 wire, 1000X.

3.3 XRD

XRD patterns of melt-spun wires of SMA-48 were made at room temperature using Cu K α radiation. Diffraction scans of three conditions are shown in

Fig. 4. X-ray diffraction scans of SMA-48 using Cu K α radiation at room temperature: (a) as-spun wires, (b) oil-quenched, (c) cold-worked wires.

Fig. 4 Scans from top to bottom are: (a) as-spun wire, (b) for comparison an alloy ingot fabricated and oil quenched from 1300°C, and (c) cold-worked wire. The peaks correspond to: (1) the L1₂ phase, (2) the tetragonal martensitic phase, (3) the Ni₅Al₃ phase, and (4) the B2 phase. Scans of the as-spun wires (a) showed that the material consisted mostly of Ni₃Al (L1₂) and NiAl (B2) phases with a small amount of bct martensite and one peak at 46° that appeared to be bco martensite. After etching to approximately half their original diameter to eliminate surface effects, similar results were found.

Scan (b) is of a sample that was ingot cast, fabricated, and oil quenched from 1300°C. In comparison to the IRL wires, no retained NiAl (B2) phase was present, and the phases were Ni₃Al (L1₂) and tetragonal martensite.

Some specimens of the as-spun wire were cold worked by forging before analysis and shown in scan (c). In this case, the L1₂ phase was still present; however, almost all the metastable B2 phase has transformed into the bct martensite phase. The relative

amounts of L1₂ vs bct appear similar to that of the ingot-fabricated and oil-quenched material.

Previous work on this series of alloys found that bco martensite was formed by conventional melt spinning [4] and that bct martensite was formed by bulk casting and oil quenching [18]. The two types appear to be associated with changes in cooling rates and small compositional differences. The presence in the IRL wires of both phases leads to the conclusion that this process results in an intermediate solidification rate.

3.4 Shape-Memory Effect

A bend recovery test was performed by bending an SMA-48 wire to an angle of 40° at room temperature then heating to 300°C at a rate of 10°C/min. The bend angle was measured as a function of temperature. Bending recovery starts immediately upon heating or cooling. A sharp recovery of the bend angle occurred at 180°C that agrees well with the A_p of 184°C determined by differential scanning calorimeter (DSC) [26] for specimens of fabricated and oil-quenched SMA-48 alloy. Samples that were annealed for 5 h at 200°C gave essentially the same results.

Some degree of two-way shape memory was seen in both cases.

Wire samples were also thermally cycled in a tensile machine to determine the shape-memory effect. Using a current of 6 A, they were heated to 250°C then cooled to 100°C in 20-s cycles. A constant load of 965 MPa was applied, and the tensile strain versus time was measured. The shape-memory effect began when the temperature reached ~180°C and the sample shortened. The length change ceased after the temperature reached 250°C. Upon cooling, the wire returned to its original length. The 0.3% strain recovery continued unchanged through 40 cycles. No indication of change occurred after 40 cycles. The maximum recovery strain of the alloy wire as a function of prior cold work has not yet been determined.

4. SUMMARY

The IRL melt-spinning method produced net-shape ductile wires of an Ni-Al-Fe-B alloy. The as-spun wires contained four phases: B2, L1₂, together with bct and bcc martensite. The B2 and bcc phases transformed to bct martensite upon cold working. Shape-memory tests confirmed an A_p temperature of 180°C.

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