CHARACTERIZATION OF HE NI-TI RIBONS SHAPE MEMORY ALLOYS (SMAs) FABRICATED BY MELT-SPINNING

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Abstract. *Ni-Ti shape memory alloys (SMAs) have received considerable research attention and are widely used because they combine special functional properties with high mechanical strength. These characteristics are due to the martensitic transformation and its reversion, which can be activated by thermal or mechanical loads. Generally there are several advantages of rapid solidification (quenching rate varies from 10^3 up to 10^7 K/s) over the slower conventional solidification techniques. These are an ability to form metastable phases, increasing the solubility above the equilibrium solubility, decreasing the segregation of additions, and refining the microstructure. It is considered that all of these effects have been attempted to improve shape memory effects in the rapidly solidified ribbons. When the ribbon is produced at a higher wheel velocity in melt spinning, the degree of supercooling becomes high because of its thinner thickness. The grain boundaries and defects can act as barriers to the martensitic transformation as a result of extra energy required during transformation.*

Keywords*: Ti–Ni alloys ribbons, shape memory alloys, rapid solidification.*

1. INTRODUCTION

Smart materials are those capable of reacting to changes in the environment that surrounds them. Among these materials are alloys with Shape Memory Effect (SME), which are metals that when subjected to a plastic deformation have the ability to return to their original shape form after a subsequent heating. According to Miyazaki et al. (1989), the first leagues to SME have been developed in the 50s in Au-Cd (1951). Ni-Ti alloys exhibit the best results among the alloys SME, with shape recovery of around 8%, at temperatures ranging from -50 º C to 110 º C (Otubo et al, 2000).

The first method developed for the production of Ni-Ti alloys was by merging in a vacuum induction furnace (VIM - Vacuum Induction Melting), which uses a graphite crucible and ingot special. That graphite is responsible for the contamination by carbon that reacts with both nickel and titanium. The oxygen contamination is due to the pressure not so high (around 10 Pa) within the fusion chamber. One solution to circumvent such contamination was the use of the process of electron beam melting (EBM - Electron Beam Melting) (Stoeckel, 1989). In this case, fusion occurs in a copper crucible cooled by eliminating the carbon contamination. As if working in a vacuum of approximately 10-2Pa, becomes minimal contamination by oxygen. The rapid solidification of alloys of NiTi system has traditionally been studied for Ti-rich compositions as compositions Ti65% at.Ni, Ni60% at.Ti (Funakubo, 1987) in 23-46% at. and 55-64% at. Ni was reported to be forming in amorphous ribbons solidified by melt-spinning. "However, no relationship between the composition Ni50% at.Ti and the formation of amorphous structure was discussed.

According to (Castro et al, 2001) There are several advantages of using rapid solidification in alloys with shape memory effect, namely: ease of forming metastable phases, sharp decrease of the problem of segregation, increased solid solubility, microstructural refinement, the possibility of obtaining glassy metals or quasi-crystalline and manufacture of thin ribbons of alloys for specific applications such as micro sensors and / or micro actuators. Therefore, the objective was to analyze the microstructure and its influence on transformation temperatures of Ni-Ti tape produced by a rapid solidification.

2. EXPERIMENTAL

2.1. Experimental Procedure

Ni-Ti alloys used in this study were supplied by Instituto Tecnologico Aeronautica (ITA) under the project "Casadinho" UFCG ITA-funded by CNPq. The ingot of NiTi-based alloy was produced via electron beam melting (EBM - Electron Beam Melting). We used a furnace model EMO80 80kW of power belonging to the Institute for

Technological Research of São Paulo (IPT). To produce the ingot used a semi-dynamic process in which the power load is made of a continuous caster and a static manner, ie, it uses a constant volume of ingot.

The dimensions of the ingots produced were 38 mm in diameter and maximum height of 50mm (height of the casting) and weight of 445g. Thus was obtained ingot with composition 55.5% Ni - 44.5% Ti (wt%), called EB5. Subsequently samples were characterized by bullion differential scanning calorimetry (DSC) with a model DSC 404C of NETZSCH, belonging to the ITA, conforn presented in Tab. 1. The methodology can be found at work (Sashihara, 2007).

Table 1: Chemical composition (wt%) and the transformation temperatures of NiTi alloy ingot (EB5).

Sample	Ni (%wt)	C (%wt)	O (%wt)	M_I (°C)	M_P (°C)	M_F (°C)	A_I (°C)	A_P (°C)	A_F (°C)
EB5	54,90	0,014	0,064	66,50	55,00	45,00	73,10	86,30	95,20

2.2. Production of NiTi ribbon

To obtain the tape was used the technique of rapid solidification by Melt Spinning. In this case we use the principle of rapid extraction of heat. Pieces of the ingot were previously prepared and placed into a crucible and those living inside an induction coil which is in a chamber with argon atmosphere and then be remelted. The ejection occurred in the liquid alloy temperature of about 1350 °C at a pressure of 200 mbar on a copper wheel in motion with rotating speed of 50 m/s. The distance from the tip ejection crucible for the steering wheel was 0.5 mm and the angle between the crucible and the vector normal to the wheel was 5, figure 1.

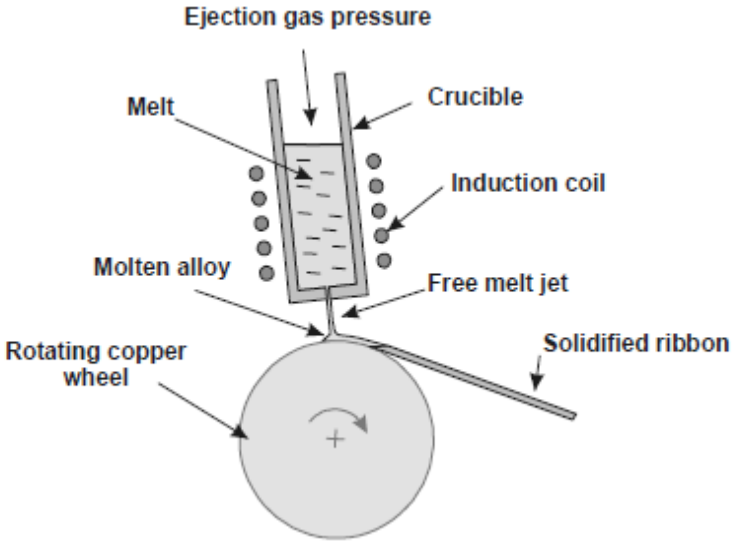

Figure 1: Melt Spinning process.

2.3. Optical Microscopy

The process of embedding the Metallographic sample preparation and manual were performed at the Laboratory of Metallography of UAEM / UFCG. The tape was embedded longitudinal thickness using acrylic resin to cure hot (VIPI FLASH) as Figure 2.

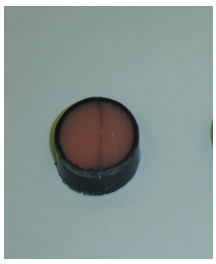

Figure 2: Ni-Ti tape - built in the longitudinal direction of the thickness.

As for the manual preparation, the surfaces of samples were ground in a manual grinder, using the following sequence of sandpaper-grained Silicon Carbide (SiC): 220, 400, 600 and 1200. Between one and another sanding, and sanding change the direction of 90 °, the samples were washed in running water for the purpose of removing waste from the previous sanding.

The polishing procedure was conducted on a manual lathe, initially with three alumina grain size (0.3 mm) and then 2 (0.05 mm). Between one and another polishing the samples were washed in tap water and dried using ethanol is assisted by an electric dryer.

2.4. Calorimetric analysis

The thermal properties of the material can be investigated through the technique of differential scanning calorimetry consists of measuring the difference between two thermal signals from two different samples, one containing the material to be studied (sample test) and the other containing a material known thermal behavior of (sample - reference). As the thermal behavior of the sample - reference is known, one can determine the thermal evolution of the test sample, which includes obtaining data such as crystallization temperatures, melting temperatures, glass-transition temperatures,

thermal relaxation regions, enthalpies involved in these processes, specific heats, etc. For the tape was observed EB5 Ni-Ti transformation temperatures.

The calorimeter is used Mettler Toledo brand, model 823e. The tests were performed at a rate of 10 ° C / min for both segments (heating and cooling) and the tests were performed in the temperature range from -30 ° C to 300 ° C.

2.5. Microstructural characterization - XRD

The technique of x-ray diffraction is based on the diffraction of x-ray photons by electrons of the atoms in the sample under study. The x-ray spectrum matches the measured intensity of the scattered beam by the material according to the angle of the beam on the sample, and this intensity depends on the constructive or destructive interference of waves diffracted by different atoms. The technique - Ray Diffraction (XRD) aided in identifying the phases of Ni-Ti ribbon.

The equipment used was a ray diffractometer Brand X SHIMADZU model XRD 6000 using Cu Kα radiation obtained from the copper target tube at a voltage of 40 kV. The sample was prepared and placed in sample port, which was fixed with double-sided tape.

3. RESULTS AND DISCUSSION

Melting spinning method with the technique, we obtained a tape called Ni-Ti EB5, with a thickness of 25μm, as observed by (Anselmo, 2010) at the same speed of the wheel covers (50 m / s). One explanation lies in the fact that the coefficient of heat transfer ribbon-wheel interface is higher and the cooling rate increases, causing a high heat extraction. This means that the amount of molten metal deposited on the wheel is much smaller when increasing the speed of rotation of the wheel, producing a thin ribbon, as is shown in Figure 3. Figure 3 shows an optical micrograph of a conventionally etched ribbon (wheel speed 50 m/s). The microstructure is primarily single phase, with small amounts of secondary phase distributed in the matrix. Since the stoichiometric range of Ni-Ti is very narrow at low temperatures, it is quite usual that the material contains precipitates of a second phase (Ti2Ni) precipitates. From Fig. 3 it seems that the grain size is about 5 -10 lm, therefore we cannot be sure that this is the real grain size.

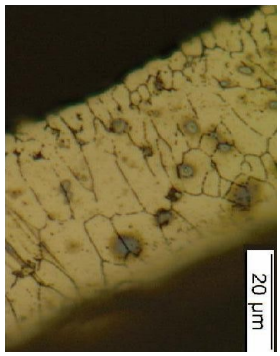

Figure 3: Microscopy of the ribbon, called NiTi EB5, with a thickness of 25μm.

Measures of Differential Scanning Calorimetry (DSC) were obtained for the tape in order to measure the transformation temperatures. Figure 3 is related to the EB5 tape where we observe two peaks of transformation in both the heating and cooling.

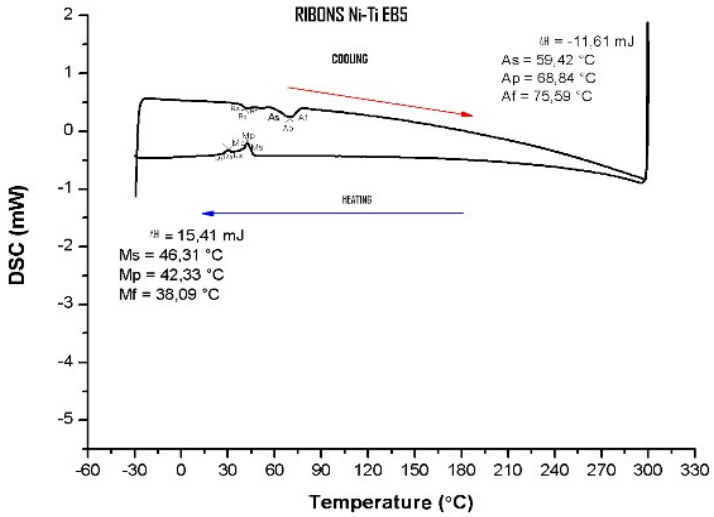

Figure 4: DSC curves of NiTi ribbon EB5.

It was observed that with the speed of 50 m / s wheel NiTi tape showed a decrease in transformation temperatures, as observed by (Anselmo, 2010) which obtained a tape NiTi transformation temperatures decreased with increasing wheel speed. This decrease in the values of transformation temperatures is related to grain size, ie, the higher the wheel speed, a smaller grain microstructure is obtained in the league, as illustrated in Figure 3..

The results of XRD experiments were obtained from the free side of the ribbons shown in Figure 5. The results also show that all of the ribbons manufactured at the different wheel speeds are crystalline and

It is observed that with increasing wheel speed, the R phase is suppressed and only the B19 phase (TiC) appears in the diffractograms presented. With the increasing speed of rotation of the wheel of 30 m / s to 40 m / s, the R phase was completely suppressed. This is probably due to the reduction of Ti in the league, because the Ti favors delaying the formation reducing the martensitic transformation temperatures (Dutkiewicj et al, 1999). The fact that the R phase is suppressed or completely disappears can probably be attributed to the increased level of super-cooling caused by increased rotational speed of the wheel that would lead to production phase metastable (Park et al, 2006). It is observed in some studies in the literature that the removal of all phases in certain regions, especially on the side of the tape in contact with the wheel, there is the fact that the formation of fully amorphous ribbons (Kima et al, 2006b; Kima et al, 2006a).

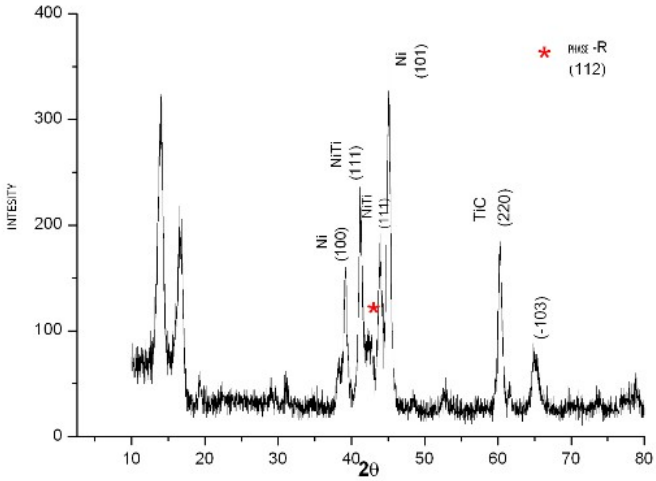

Figure 5: XRD pattern of NiTi Tape EB5.

4. CONCLUSIONS

After the results obtained in this study reached the following conclusions:

1. The melting spinning technique allows the production of tapes of about 30 μm in one processing step;

2. The work of this paper has shown that the melt-spin technique is able to produce samples with good shape memory properties immediately after cooling even without subsequent heat treatment.

3. The higher the wheel speed, the smaller the thickness of the resulting ribbon and therefore the higher the cooling rate. With increasing cooling rate the microstructure gets finer and finer and therefore metallographic preparation was not easy at all.

4. The material with very fine microstructure has also good mechanical properties (strength and ductility) and therefore exhibits good potential for future microactuator applications.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

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