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# Load-biased martensitic transformation strain of Ti<sub>50</sub>-Ni<sub>47</sub>-Co<sub>3</sub> strip obtained by a twin-roll casting technique



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#### ABSTRACT

Mechanical properties of the  $Ti_{50}$ – $Ni_{47}$ – $Co_3$  (at%) strips produced by twin-roll casting are analyzed, focusing on the maximum recoverable strain obtained by inducing the martensitic transformation under constant tensile load. The recoverable strain achieves a value close to 5.5% for a load of  $\sim$  120 MPa. The microstructure shows a typical cellular solidification structure, where the austenite grains tend to a columnar array morphology with strong  $\{100\}_{B2}$  fiber texture aligned with ND (perpendicular to the strip surface). On the basis of the measured texture, the maximum B2 $\rightarrow$ B19′ transformation strain was estimated at 5.6%, using a Sachs-type upper bound model, which averages the most favorable martensite variants in each grain, disregarding interactions between them. The agreement between the measured maximum recoverable strain and the model's outcome is ascribed to special properties of Ti–Ni alloys, which allow them to overcome incompatibilities in shape changes of transforming grains in this particular polycrystalline environment.

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#### 1. Introduction

Ni–Ti shape memory alloys (SMA) are recognized due to their remarkable functional and physical properties, such as large recoverable strains, corrosion resistance and biocompatibility. This system is nowadays the most widely used in the biomedical field. For some specific applications, the transformation temperatures, hysteresis, and relative stabilities of the phases involved in the underlying martensitic transformation must be modified. This has led to the exploitation of different tools of the physical metallurgy, and in this work we make use of two of them: the addition of a third element and the use of rapid solidification non-conventional production technique.

A small amount of Co is introduced in order to promote the separation of the B2 $\rightarrow$ R and R $\rightarrow$ B19' transformations, due to a shift of  $M_s^{(B19')}$  downwards lower temperature range [1,2], which is more appropriate for some dental and medical applications [3]. On the other hand, the twin-roll casting (TRC) is a powerful technique, which allows us to obtain semi-finished strips in a one-step procedure directly from the melt. Moreover, a favorable microstructure with a columnar grain morphology and a small grain size can be achieved [4–6].

It is known that the texture plays a very important role in determining the recoverable strains of polycrystalline shape memory alloys [7]. Rapid solidification produces strong textured columnar grains, where  $[001]_{B2}$  is parallel to a normal direction, and then the strip presents a mixture of  $[100]_{B2}$  and  $[110]_{B2}$  directions, in length and width [8] (directions of type  $[uv0]_{B2}$ , distributed between  $[100]_{B2}$  and  $[110]_{B2}$ , lie parallel to the strip plane). This orientation distribution could mean a priori certain drawback in such polycrystalline aggregate in order to maximize the transformation strain of a Ti–Ni based alloy. Here, it is worth noting that for the  $B2 \rightarrow B19'$  transition the maximum value is achieved for the  $\langle 355 \rangle_{B2}$  directions (above 10%) and directions clustered around, while lower values are obtained for directions spreading between  $\langle 100 \rangle_{B2}$  and  $\langle 110 \rangle_{B2}$  (between  $\sim 4\%$  and  $\sim 9\%$ ).

The results reported in the literature remain somewhat controversial: Shu and Bhattacharya [9] evaluated the inner bound of the transformation strain, imposing the same strain on each grain, assuming a strong constraint between neighboring grains. These authors recommend this inner bound as a reasonable estimation of the recoverable transformation strain. Under these hypotheses, the recoverable strain was predicted around 2.3% in Ti–Ni alloys with {001}<sub>B2</sub> fiber texture.

In Ti–Ni twin-roll strip Goryczka and Ochin [10] reported a recoverable experimental strain of about 3%. Similar values of strain were measured in a Ti–Ni–Co<sub>3</sub> twin-roll strip [11]. In melt spun Ti–Ni ribbons with this texture, Eucken and Hirsch [12] reported that a reversible strain reach 4%. However, most recently [5,13], recoverable strains between 5% and 6% were observed in Ti–Ni–Cu<sub>5</sub> and Ti–Ni meltspun ribbons respectively.

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For additional insights into the factors involved in the recoverable transformation strain in the strong textured Ti–Ni alloys, we analyzed a Ti<sub>50</sub>–Ni<sub>47</sub>–Co<sub>3</sub> strip produced by twin-roll casting. The recoverable transformation strain measured in load-biased thermal cycling through the martensitic transformation was compared with upper bound of the value predicted by a simple model. This model averages the strains of the constituent grains disregarding their interactions, and taking into account the texture of the rapid solidified strip by introducing the inverse pole figure as a weight function.

#### 2. Experimental set-up

The twin-roll casting consists in the solidification of a melted alloy between two symmetrical rollers, which produces a continuous solid sheet. These rollers rotate at high speed in opposite directions. This system is located in a vacuum chamber in a purified helium atmosphere to protect the material from oxidation and improve thermal conductivity inside the chamber. The melted alloy, placed in a quartz nozzle, is ejected by supplying a pressure of argon, into the nip of the rollers. The processing parameters used in the production of the Ti<sub>50</sub>–Ni<sub>47</sub>–Co<sub>3</sub> strip are given in Table 1. For detailed description of the twin roll casting procedure applied to Ti–Ni based alloys see Refs. [10,14].

The transformations were characterized by a Mettler DSC 30 differential scanning calorimeter. The electrical resistivity behavior was characterized in a.c. mode, using a generic function generator and a Sr-530 Lock-in amplifier. Strip microstructure was observed using an Olympus PME3 optical microscope and a FEI TECNAI F20 transmission electron microscope (TEM). The specimens were mechanically polished up to 1 µm diamond paste and etched with 1HF+4HNO<sub>3</sub>+5H<sub>2</sub>O solution for the optical microscope analysis. The thin foils examined in the TEM were prepared with a double jet using 95% acetic acid and 5% perchloric acid. The pole figures were recorded with a Philips X' Pert Pro MPD diffractometer. equipped with a Cu tube, X-ray lens optics, parallel plagues and a graphite monochromator on the outcoming beam and an Eulerean Cradle for texture measurements. Measured pole figures were corrected for defocusing and background and further analyzed by WXpopLA to obtain orientation distribution functions and guarantee pole figure compatibility.

Load-biased thermal cycling was performed in a custom-built machine, which allows us to apply a constant load and program cooling/heating cycles. The machine has an Instrom load cell (5 kN) to measure the stress. The strain is measured by an *Epsilon* 3542 axial extensometer with 10 mm gauge length, clipped between the specimen grips. The sample was cooled with nitrogen vapor, so it was necessary to enclose the sample and the grip in an acrylic cylindrical chamber. Two electrical tubular cartridge heaters inserted into drilled holes of the grip body were used for heating. The system sketch is shown in Fig. 1.

The temperature was measured by a K-thermocouple located in the center of the sample. Besides, the sample was isolated with alumina wool, in order to be cooled and heated by temperature

**Table 1** Twin-roll casting parameters.

Material	Ti <sub>50</sub> Ni <sub>47</sub> Co <sub>3</sub>
Melt temperature (K)	1783
Roller material	Cu-Co-Be
Rollers speed (m s <sup>-1</sup> )	0.6
Ejection pressure (mbar)	250

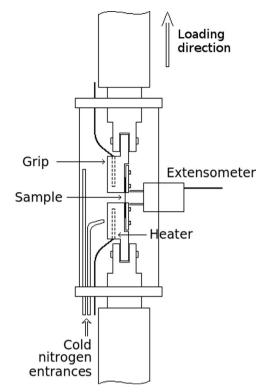


Fig. 1. Thermal cycling load-biased system.

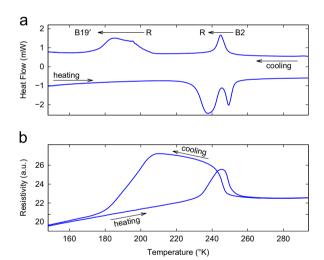


Fig. 2. DSC (a) and resistivity vs. temperature (b), cooling and heating curve of the  $\text{Ti-NiCo}_3$  strip.

conduction from the grips, keeping the temperature uniform through the assembly of a lower grip-sample-upper grip.

#### 3. Results

### 3.1. Thermal-induced transformation and characteristic temperatures

A DSC measure of the as-cast strip is presented in Fig. 2(a). The  $Ti_{50}$ – $Ni_{47}$ – $Co_3$  strip undergoes two subsequent martensitic transformations, the first from the B2 parent phase to rhombohedral R-phase and the second transformation between R-phase and monoclinic B19′. The R transformation has small temperature hysteresis

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