ANALYSIS OF THE EFFECT OF TEMPERATURE ON HOT TENSILE TESTING IN NiTi ALLOY*

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Abstract

. In this work, samples of the alloy with composition of 50.9 % at. Ni were machined and were subjected to hot tensile test, after the machining all the samples were solubilized so that all were in the same condition during the hot tensile test. The hot tensile tests were performed between the temperatures 350-650°C and the yield stress were between 800 and 597.1 MPa and area reduction between 17 and 75% with respect to the initial area. The test at 350°C shows a fragile fracture characteristic while the hot tensile test above this temperature showed a predominantly ductile fracture. The fragile fracture is evidenced by the river marks that are characteristic of this type of fracture, because in this condition does not promote much plastic deformation. The higher temperatures present ductile fracture, because present the dimples that also characterize this type of fracture. This work shows the temperature relationship in the mechanical properties, more specifically the hot tensile test, in the NiTi alloy.
Keywords: Shape Memory Alloys; NiTi alloys; Hot Tensile Test;

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1 INTRODUCTION

The approximately equiatomic alloy composition of nickel and titanium (NiTi) is known since the 1960s through Willian F. Buehler and his collaborators [1]. In Brazil, despite the great applicability of the alloy, alloy processing began only in the 90's due to the difficulty of manufacturing due to oxygen and carbon contamination in the production and subsequent thermomechanical processes [2–4].

The NiTi has a stand out among of shape memory alloys because it presents better results with respect to shape recovery than other alloys that have the same property [5–7]. In addition, the NiTi alloy presents excellent fatigue resistance, biocompatibility and corrosion resistance (8.9). Due to these excellent properties NiTi alloy stands out in the most varied applications as aerospace, automotive, naval, robotics, biomedical, civil and nuclear [8,9]

The properties of the NiTi alloy are highly dependent on the chemical composition of the material, for example, the variation in the Ni / Ti ratio for alloys with 50.6% at. Nickel solubilized and subsequently aged by heat treatment promotes the formation of coherent precipitates rich in nickel [7,10]. The alteration in the ratio of the elements in the matrix phase drastically affects the transformation temperature of the alloy; for example, variation of 0.1% atomic in the nickel concentration will cause to a decrease of approximately 10 °C in the alloy transformation temperature [4,11].

The temperature of the tensile test influences the phases present in the material and, consequently, modify the mechanical properties of the alloy [12]. Furthermore, plastic deformation for a material below the recrystallization temperature will cause an increase in the mechanical strength of the alloy depending on the test temperature and may result in fracture of the material [13–15].

The hot tensile test is important for determine parameters such as area reduction, elongation, yield stress at the temperatures tested. In addition, the results of hot tensile test are important for determine the fracture mechanisms in function of temperature, because this data are important, for example for the mechanical conformation processes as a function of temperature [14,16,17].

The objective of this work is to analyze the results of the hot tensile test at 350 ºC, 450 ºC, 550 ºC and 650 ºC of the NiTi alloy and to relation the data of mechanical properties and the mechanisms of fracture at the temperatures of the test.

1. EXPERIMENTS

2.1. Materials and conformation mechanics

The sample NiTi with composition of 50.9 % atomic nickel and initially the bar about 15 mm in diameter. This bar was mechanically conformed by forge rotative FENN 5F the according to the hammers the shown in Table 1:

Table 1 - Values of the hammer diameters used in hot forging

Pass	Forge 5F hammers 4 φ (mm)	Reduction in Area (%)
1	13.843	14.83
2	11.938	25.63
3	10.414	23.90
4	9.525	16.34

The bar was reduced by hot forging with heating the bar in the Hevi-Duty 18kW (1370 °C) muffler furnace to 850 °C for 15 minutes. This process mechanical was until the bar reach diameter approximately 9.5 mm so that it can be used next stages.

2.2. Machining and heat treatment of the sample

The samples were machined as required by the standard ASTM E08 and ASTM E04 ([18,19]). The samples were encapsulated in a quartz tube in an argon atmosphere and annealed at 850 °C for one hour in the 18kW Hevi-Duty furnace this heat treatment was used to solubilize the sample, because in this state the microstructure will be a supersaturated solid solution free of precipitates, so that all samples start with the same microstructural characteristics.

2.3. Hot tensile test

The samples after solubilization were conducted for the hot tensile test at temperatures of 350 °C, 450 °C, 550 °C and 650 °C. The tensile tests were conducted in the hot tensile machine INSTRON 3382 with load cell 100 kN, oven reaches temperatures to 1200 °C, at strain rate is 0.5 mm / min and the data acquisition software is Instron serie IXTM / s as specified in ASTM E21 [20].

2.4 Scanning electronic microscopy (SEM) and Transmission electronic microscopy (TEM)

The fractures of the samples of the hot tensile were analyzed by scanning electron microscopy (SEM) to better understand the fracture mechanisms of the material. The equipment used was the scanning electron microscope TESCAN model VEGA 3 XMU.

2. RESULTS

The tensile test hot in NiTi alloys is necessary due to the growth in the number of applications in various areas. For the success of the mechanical conformation and the aging test is necessary determine important parameters such as yield stress, elastic modulus, stress, maximum strain of the NiTi alloy. Figure 1 shows the stress graph (σ) versus deformation (ε) in the samples hot tested.

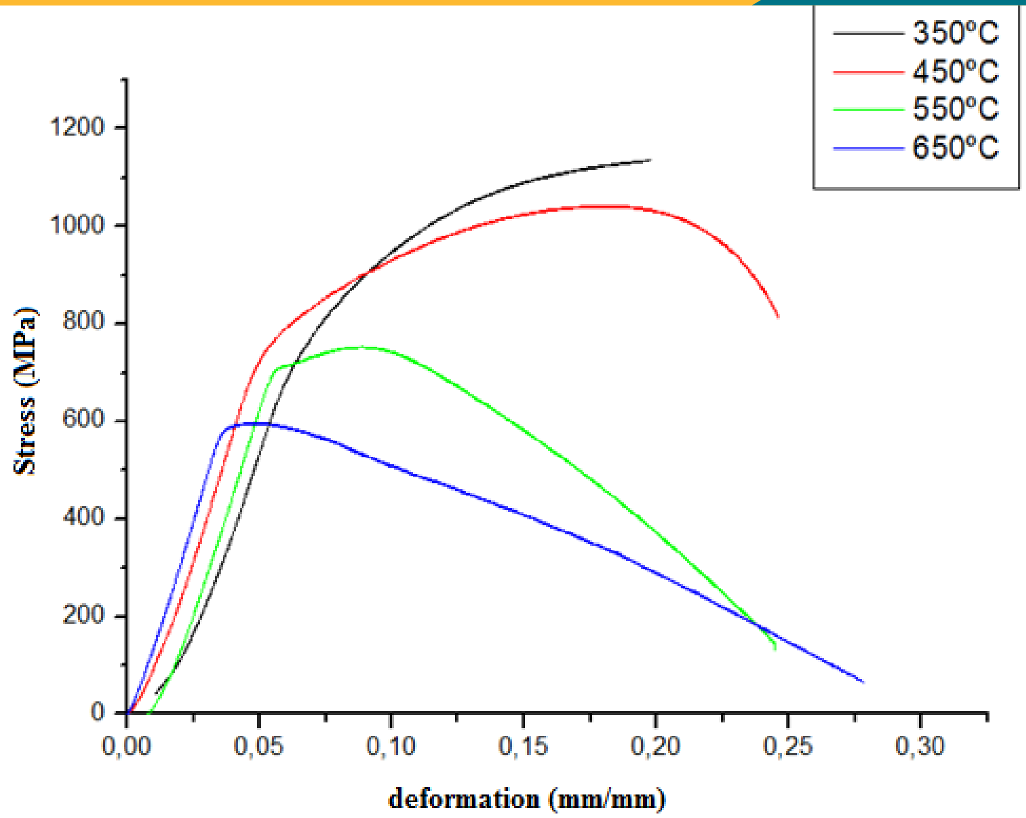

Figure 1 - Hot tensile stress – strain curve for NiTi alloy

The table 2 shows the values of mechanical properties of the NiTi alloy after the hot tensile test:

Table 2 - Mechanical properties of the NiTi alloy after the hot tensile test.

Temperature (ºC)	Yield Stress (MPa)	Deformation (%)	Reduction Area (RA - %)
350	800	19.5	17
450	750	24.4	39
550	720	24.5	56
650	597	27	75

In table 2 show the results of sample tested at temperature of 350°C with yield stress was approximately 800 MPa and 17% of reduction area compared to initial area of the sample. The figure 1 showed the sample fractography of NiTi alloy after the hot tensile test to 350 °C with cleavage fracture characteristic.

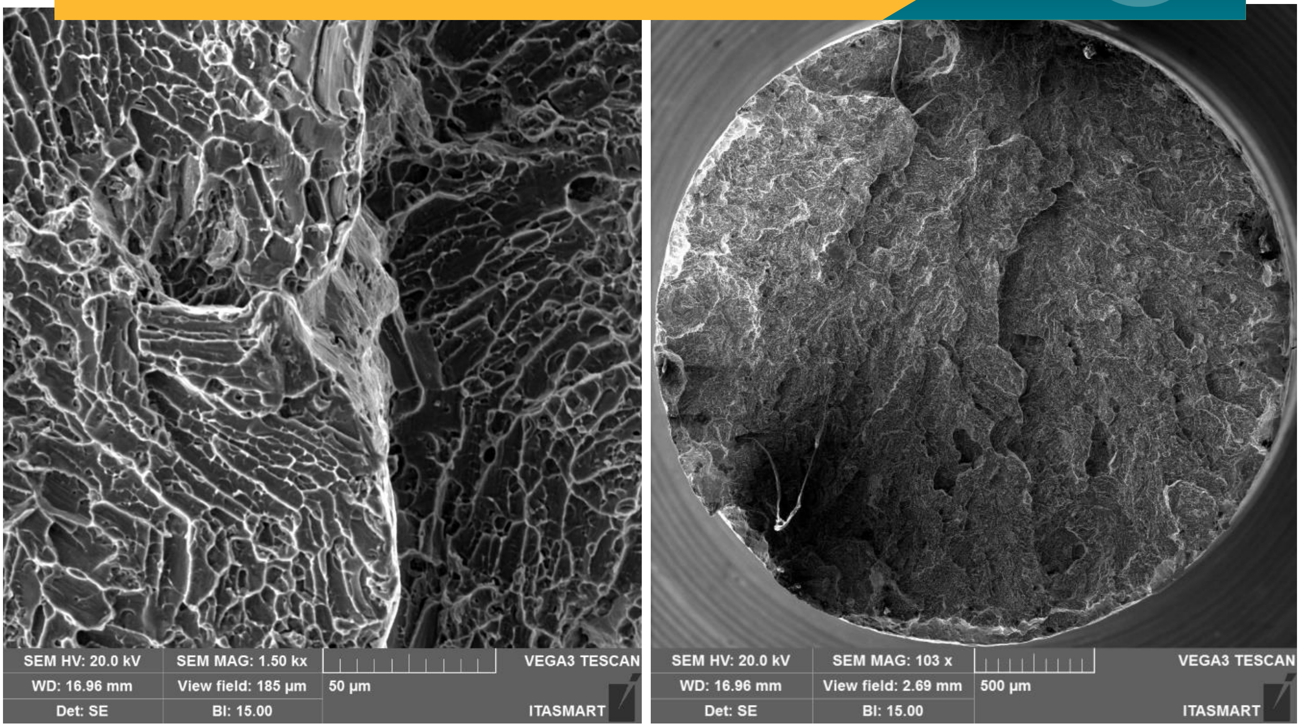

Figure 1 - Fractography after the hot tensile test at a temperature of 350 ºC

The transgranular fracture has cracks pass through the grains and your fracture surface is faceted due to different orientations of the cleavage planes (cleavage facets). The fragile fracture is approximately perpendicular to the applied stress and produces a relatively flat, shiny surface. The crack propagation is through crystalline planes separation of the metal, preferably the cleavage planes or those with higher probability of cleavage fracture, as well as fracture surface shown in figure 1.

The cleavage fracture normally occurs in materials having body-centered cubic structure, hexagonal close-packed structure (hcp), ionic crystals, covalent crystals and finally under specific conditions (high strain rates and low temperatures) for the face-centered cubic structure. The cleavage surface is marked by a relief called "*river marks*". These river marks arise with growth of the cleavage facets in parallel planes.

The table 2 show sample tested at a temperature of 450 °C that results in yield stress is approximately 750 MPa and reduction in area of 39% compared to the area of the sample at the before starting of hot tensile test. Besides that, the fracture of the sample NiTi after the hot tensile test in this temperature shows a characteristic of ductile fracture according to figure 2. It is observed in figure 2, also the presence of "*dimples*" formed during the hot tensile test process.

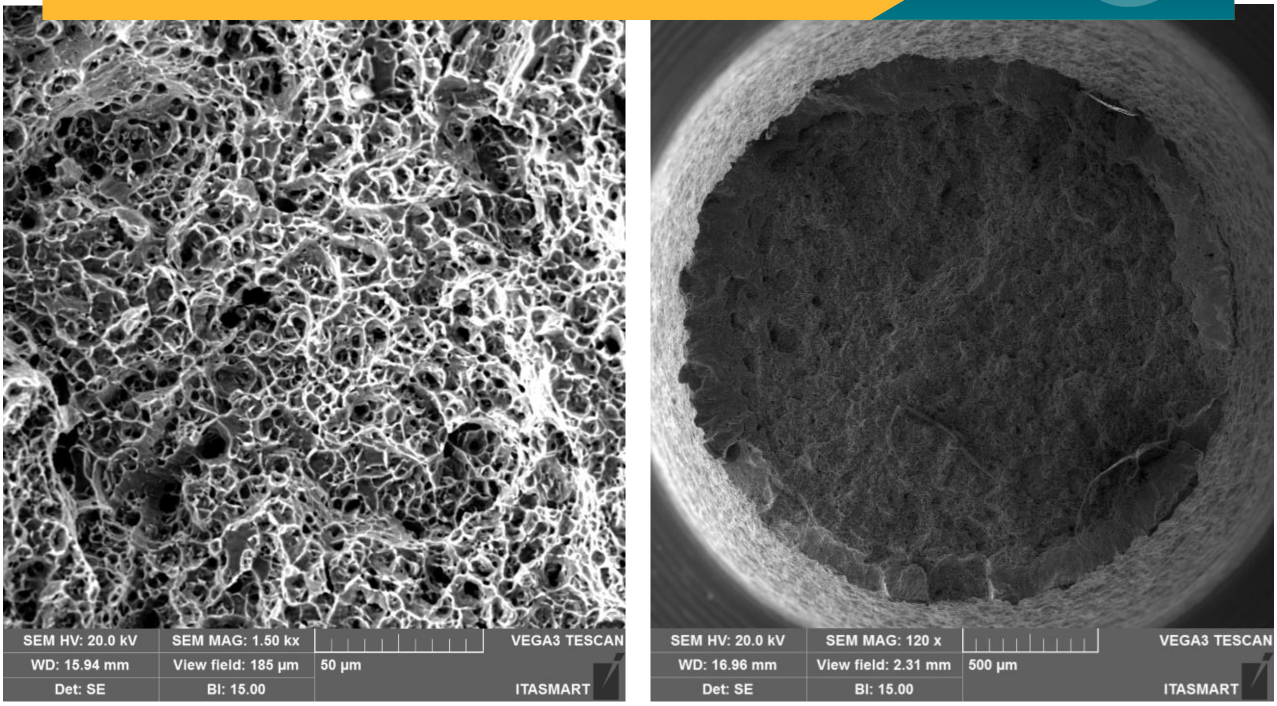

Figure 2 - Fractography after the hot tensile test at a temperature of 450 ºC

The results presented in table 2 show that yield stress of the hot tensile test at a temperature of 550 ° C was 754.20 MPa, that is, the value abruptly decreased compared to the yield stress of the alloy tested at a temperature of 450 °C. However, the fracture characteristic in hot tensile test at a temperature of 550 ° C is the same of alloy temperature at 450 ° C, in other words, the material exhibits ductile characteristics, as shown in figure 3, and with a reduction of area of 56% compared to the sample area at the beginning of hot tensile test.

The collapse of ductile materials is a rare phenomenon unlike the collapse of fragile material that uses a hypothesis in which the existing defects in a fragile material are pre-nucleated. But the hypothesis is not sustained in ductile materials, because any pre-existing discontinuity will initially be rounded by the action of plastic deformation which finishes by reducing the stress concentration at the crack. In the place of concentration of tension arises a zone of plastic deformation, due to the application of stress on the sample that tends to spherical shape. This micromechanics are given by the nucleation, growth and coalescence of microcavities. Ductile fracture has higher energy consumption during fracture than fragile fracture, which is desirable in mechanical components.

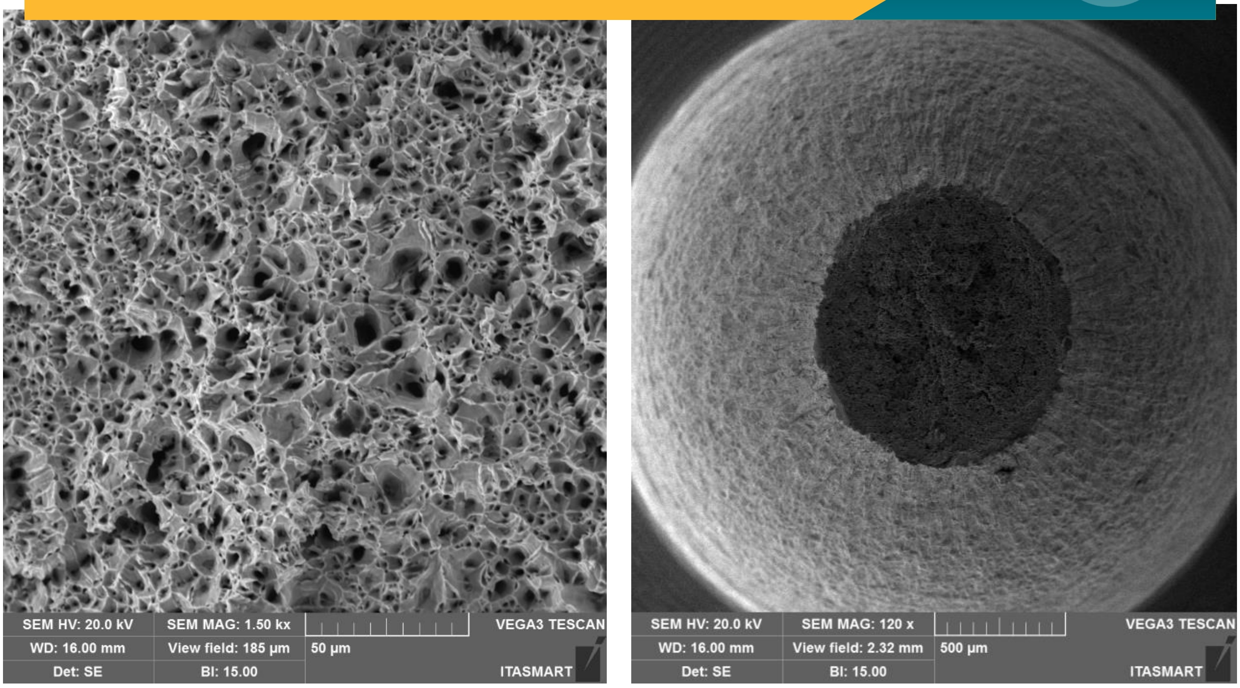

Figure 3 - Fractography after the hot tensile test at a temperature of 550 ºC

The table 2 show yield strength of the hot tensile test carried out at a temperature of 650°C was 597.10 MPa and reaching the lowest value of the yield stress of all the hot tensile tests performed in this work. The characteristic of the fracture of the alloy in the temperature of 650 ° C is extremely ductile, because the material showed a reduction of area of 75% associated with dimples formed in the hot tensile test at the temperature of 650 °C. The figure 4 show a ductile fracture surface with rough and gray appearance of the sample at temperature of 650 ºC

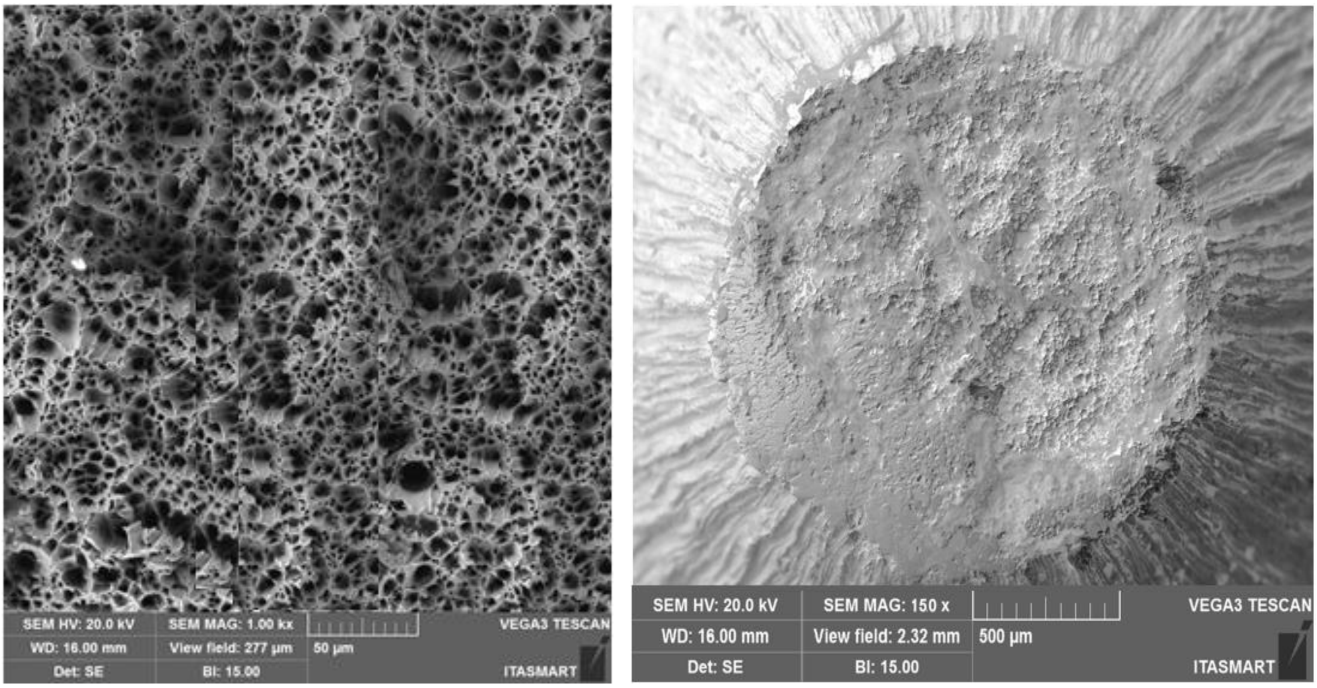

Figure 4 - Fractography after the hot tensile test at a temperature of 650 ºC

In the hot tensile test, the microcavity acquire spherical shape and the increase in tension is accompanied by increase curvature radius of the dimples, that is., in general, growth the dimples it is accompanied to an increased stress concentration. The increase in the temperature of the test also increases in the perception of mechanisms of deformation and fracture that use the relative movement of grain boundary and movement of crystalline defects during the plastic deformation.

3. CONCLUSIONS

The results show that:

- The material tested between 350 ºC and 450 ºC shows a alteration in the mechanical behavior, because below 350 ºC the material presents fragile characteristics while above 450 ºC it presents ductile characteristics;
- The evident characteristic after hot tensile test in the material with fragile fracture are the river marks and low plastic deformation;
- The evident characteristic after hot tensile test in the material with ductile fracture are the dimples and high plastic deformation;
- The increase in the temperature also increases the mechanisms of deformation and fracture that use the relative movement of grain boundary and movement of crystalline defects during the plastic deformation, for example, the test temperature of 650 ºC is relatively high compared to the melting temperature of the NiTi, because facilitates the process of diffusion and annihilation of defects and it's observed lower mechanical resistance.

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