Theme: Shape Memory Alloy

THE EVOLUTION OF INTERNAL VOLUMETRIC DEFECTS IN THE WIRE DRAWING PROCESS OF NiTi*

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Abstract

NiTi shape memory alloys (SMA) have been widely used as construction materials due to their shape memory and superelastic properties. Usually the application of the NiTi alloy is in the wire form, which is ideal shape to make springs, orthodontic arc-wire and endodontic files. Since our research group (ITASMART) started producing NiTi wires, it was noticed that NiTi SMA produced by Vacuum Induction Melting (VIM) presents carbon contamination, which is due to the melting process. Therefore, alloys rich in carbon remove titanium from matrix, forming precipitates of TiC (titanium carbide). Also, Ti_2Ni precipitation removes titanium from the matrix and these two precipitates TiC and Ti_2Ni can produce internal volumetric defect in the wire drawing process. Seeing the importance of NiTi wires as base of many applications, this work studies the formation of volumetric defects caused by precipitates in the wire drawing process of a NiTi wire drawn down to 0,39 mm in diameter.

Keywords: NiTi SMA; TiC precipitate; Ti_2Ni, Wire drawing.

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1 INTRODUCTION

Shape Memory Alloys (SMA) have applications in various fields of human activity: aerospace, automotive, nuclear, robotics, medical and others [1-6]. It is considered a strategic material and its import is severely hampered by technology holder’s countries. Thus, developing national technology both production and forming as well as tailor made applications of the alloys is critical to the country's development in the area. Today ITASMART group (acronym for ITA - Shape Memory Alloys Research and Technology) have mastered the technology of manufacturing the alloy NiTi, not by one but by three processes (Vacuum Induction Melting – VIM; Vacuum Arc Remelting under inert atmosphere – VAR and Electron Beam Melting – EBM). By the time the ITASMART produced approximately 70 ingots with different compositions of nickel/titanium, carbon and oxygen, with masses ranging from 100 g to 22 kg [7-19]. Among various existing applications for NiTi SMA, highlight applications using wire, were the material can be readily deformed by applying an external force, and will contract or recover to its original form when heated beyond a certain temperature either by external or internal heating (Joule heating). Therefore, straight SMA wires are more advantageous due to the optimal use of material (i.e. more work generated from a minimal amount of SMA material) and the loading in tension configuration [20].

NiTi wires can be obtained through the drawing process, which allows to obtain wires with long length and constant cross-section, with controlled mechanical properties [21]. In order to obtain a wire of NiTi with controlled mechanical properties is very important to control the chemical composition of the alloy in terms of contamination of carbon. Previous works, undertaken by ITASMART, showed that the carbon contamination is inherent to VIM process due to melting in a graphite crucible and highly dependence on the quality of the graphite as well as the size of the crucible used [11-16]. Therefore, alloys rich in carbon remove titanium from matrix, forming TiC precipitates (titanium carbide) and as a consequence, it changes the wire thermomechanical properties.

Contributes to titanium removal of the matrix the Ti_2Ni precipitate formation, where the titanium is removed from the matrix in a 2:1 ratio. The Ti_2Ni is a fragile precipitate, thus tends to form a fragmented and elongated precipitate in the wire drawing direction [22-24]. Seeing the importance of NiTi wires as base of many applications, this work studies the formation of volumetric defects caused by precipitates in the wire drawing process of a NiTi wire.

2 MATERIAL AND METHODS

The starting material was a wire of 1.00 mm in diameter produced by ITASMART. The wire was cold drawn to 0.398 mm with 15% of area reduction per pass, interspersing annealing at 750°C for 3 minutes.

The drawing was made in a monobloc wire drawing and heat treatments annealing between passes in a Havi Duty furnace at the ITASMART facilities, Figure 1.

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Figure 1. Monobloc wire drawing and Havi Duty furnance (from the left to right).

A set of dies with tungsten carbide core and as a grease lubricant molybdenum disulfide (MoS_2) was used in drawing. The Table 1 shows the values for dies used in each drawing pass and the Figure 2 shows the wire with 0.398 mm.

Table 1. Set of dies.

Drawing pass	Diameter (mm)
1st	0.990
2nd	0.910
3rd	0.830
4th	0.760
5th	0.700
6th	0.640
7th	0.590
8th	0.540
9th	0.490
10th	0.398

The metallographic samples were cut with refrigerated diamond saw with controlled load in order to minimize residual stress and also to avoid mechanically induced martensite. Then the samples were molded in Bakelite, grinded down to 1200 grit sandpaper with automatic grinding/polishing machine (Allied Metrep3).

To analyses the microstructure the samples were etched with a solution of 50ml H_2O + 40ml HNO_3 + 10ml HF for a period of 15 seconds. The microstructure of the samples was observed and analyzed with scanning electron microscopy – SEM (Tescan, model Vega 3 XMU), energy dispersive spectroscopy – EDS (Oxford Instruments, model X-Act SDD EDS detector) and AZtec EDS analysis software (Oxford Instruments).

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Figure 2. Wire of NiTi with 0.398 mm in diameter.

3 RESULTS AND DISCUSSION

The Table 2 shows the values found for the overall chemical composition of the alloy used to produce the wire.

Table 2. Chemical composition of the alloy NiTi and its MTT.

Ni (%at)	C (%wt)	Ms (°C)	Mp (°C)	Mf (°C)	As (°C)	Ap (°C)	Af (°C)
49,42	0.066	62.5	51.2	38.0	65.3	81.9	91.0

The analysis matensitic transformation temperatures (MTT) of the NiTi alloy shows that the alloy is in the martensitic phase at room temperature. Where, Ms (start of temperature transformation of direct martensitic), Mp and Mf (final temperature of the direct Martensitic Transformation - MT) are above room temperature. Even temperatures of reverse MT such as As (temperature at the beginning of the reversion of martensite to austenite), Ap (peak of temperature of the reversion of martensite to austenite) and Af (final temperature of the reversion of martensite to austenite) are above room temperature.

As we can see in Table 2 the alloy has high carbon content (0.066%wt) and as result the carbon remove titanium from matrix, forming precipitates of TiC (titanium carbide), in the Figure 3 it is possible to see the results for the EDS measurements of the wire with 0.39 mm in diameter.

The upper ward of Figure 3 shows the shift in chemical composition to the linescan measurement, this change in chemical composition clearly indicates that the precipitated formed is TiC. Thus, the maps in the bottom of the Figure 3 confirm the presence of titanium and carbon in the precipitate, in other words TiC, where the region in black of the maps indicates lack of chemical elements.

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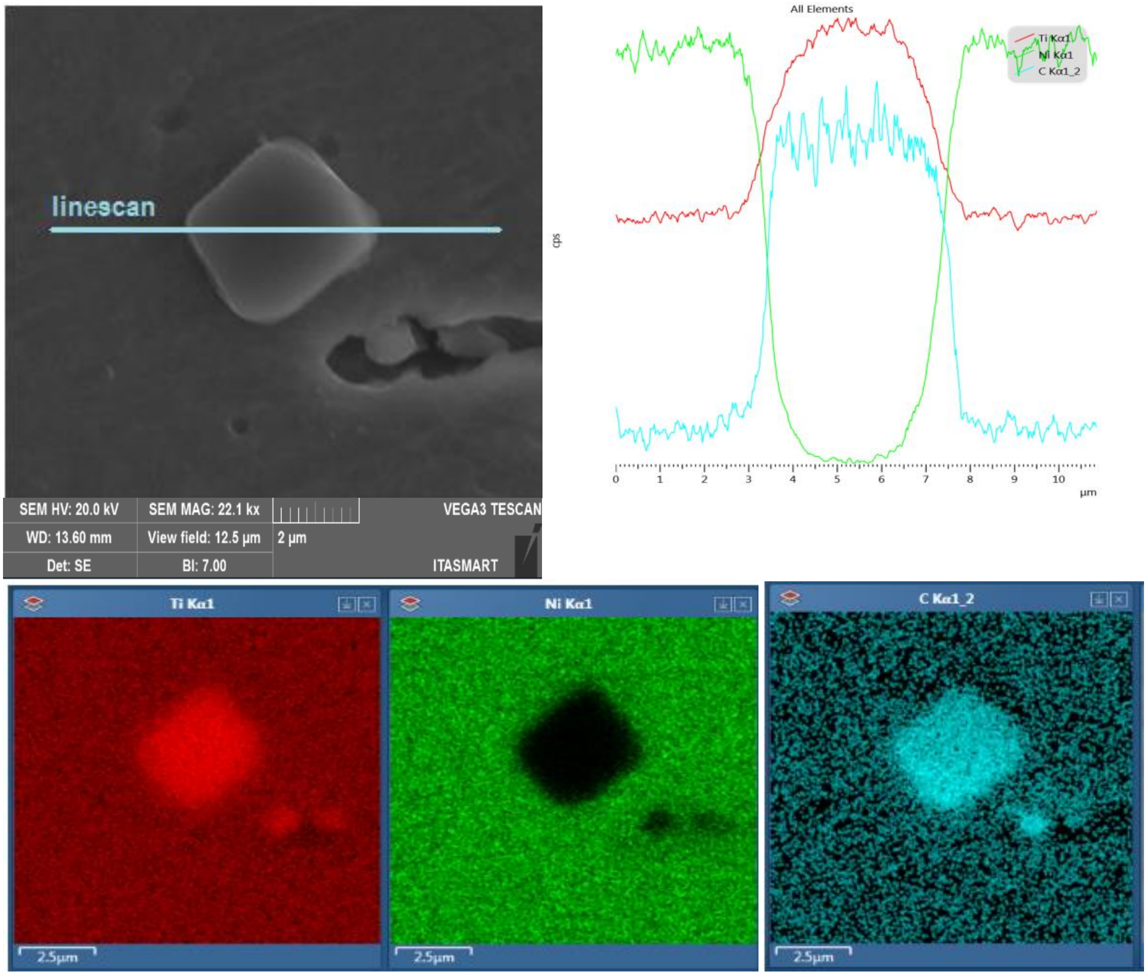

Figure 3. EDS analyses of NiTi wire with 0.398 mm in diameter.

The Figure 4 shows EDS point identification analyses for the TiC and Ti_2Ni precipitates in the matrix.

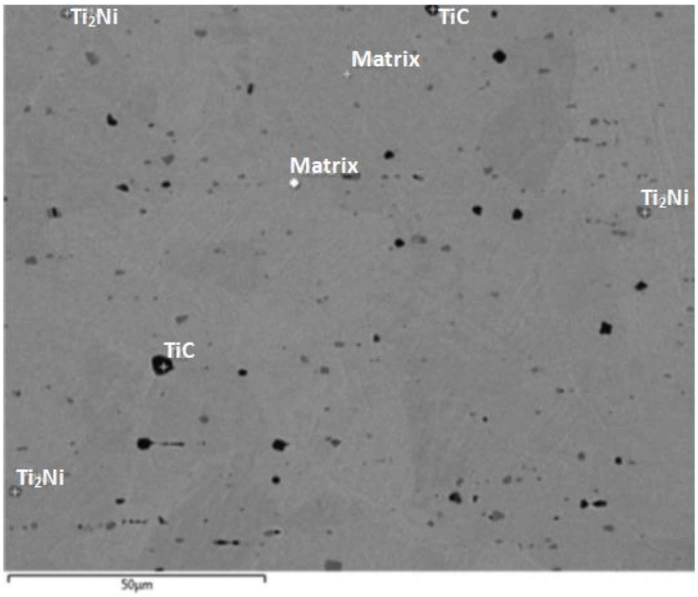

Figure 4. EDS point identification analyses for the TiC and Ti_2Ni precipitates.

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The Figure 5 shows micrographs taken in the SEM of wires with 1,00 mm and 0.398 mm in diameter, respectively. The micrographs were taken with the horizontal direction coinciding with the direction of drawing.

By the analyses of the micrographics of Figure 5 is possible to observe that precipitates in wire with 1.00 mm in diameter are distributed more widely spaced from each other along the wire if compared to distribution of precipitates from the wire with 0.398 mm in diameter. Therefore, as we can see by the micrographic, the precipitates of wire with 0.398 mm in diameter are more clusters along the wire.

One can note that the precipitates are aligned in the longitudinal direction of the wire showing the deformation texture and the reduction in diameter from 1.00 mm to 0.398 mm causes a significant increase in the trail left by precipitates along the drawing direction. This result suggests that while the wire is wire drawing to smaller diameters, there is an increase in formation of volumetric defects inside the wire.

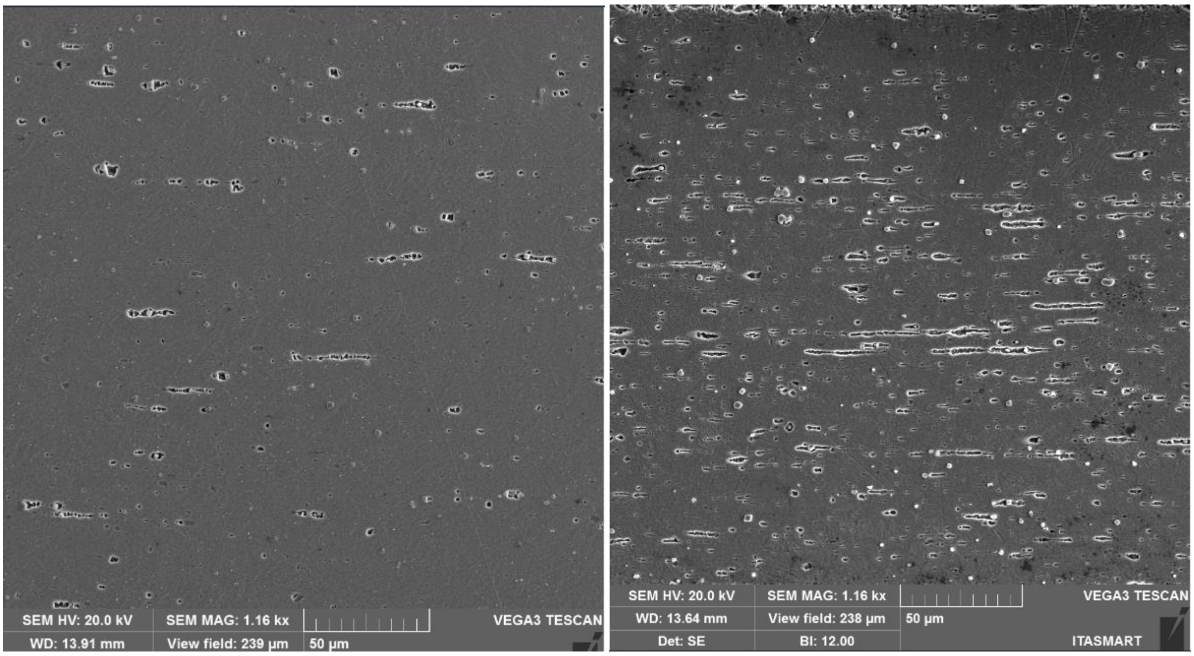

Figure 5. Micrographs taken in the SEM showing: NiTi wire with 1.00 mm in diameter (left); NiTi wire with 0.398 mm in diameter (right).

4 CONCLUSION

Wires produced by NiTi alloys with high contents of carbon show precipitates that tend to cluster inside the wire by the wire drawing process. Nevertheless, as much as the wire is wire drawing to smaller diameters the precipitates tend to align in the direction of drawing.

We notice that along the wire drawing direction occurs the fragmentation of Ti_2Ni precipitates, leaving a trail of volumetric defect inside the wire.

The amount of volumetric defects inside the wire of 1.00 mm in diameter is smaller if compared to the amount found to wire with 0.398 mm in diameter. Therefore, it is possible to infer that the more is the number of wire drawing passes in a NiTi wire with high amount of precipitates, the more is the amount of volumetric defects produced inside the wire.

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