Association of Metallurgical Engineers of Serbia
AMES
Scientific paper
UDC: 669.245'295:621.315.554]:620.18

METALLOGRAPHIC SAMPLE PREPARATION OF ORTHODONTIC Ni-Ti WIRE

Lidija Zorko*, Rebeka Rudolf

University of Maribor, Faculty of Mechanical Engineering, Smetanova 17, SI – 2000 Maribor, Slovenia

Received 30.09.2009
Accepted 22.12.2009.

Abstract

Shape memory alloys (SMA) has been at the forefront of research for the last several decades. In this field especially, Nickel-Titanium (Ni-Ti) alloys have been found to be the most useful of all SMA. The most important applications of SMA Ni-Ti alloys are namely in medicine and dentistry, where they are used as orthodontic wires. In this paper we describe the procedure of preparing metallographic samples of typical orthodontic Ni-Ti wires which are nowadays used in dentistry praxis. We prepared the samples for microstructure observation using a light microscope. Special attention is given to the metallographic preparation, which could result in damages and deformations of the sample surfaces if the procedure is incorrect. Finally, we illustrated the typical metallographic recipe for Ni-Ti SMA alloys for optical observation.

Key words: shape memory alloys, Ni-Ti wire, metallographic preparation, microstructure

Introduction

Shape memory alloys (SMA) have been an interesting topic of research for the last several decades and they refer to a unique class of materials with the ability to return to a predetermined shape when the temperature is increased. In the early 1960s, at the U.S. Naval Ordnance Laboratory the shape memory effect was discovered in an equiatomic alloy of nickel and titanium. This alloy was named Nitinol. Nickel-titanium alloys have been found to be the most useful of all SMAs. Ni-Ti SMA can exist in two different temperature-dependent crystal structures (phases) called martensite (lower temperature) and austenite (higher temperature or parent phase When the material is in its martensite form, it is soft and ductile and can be easily deformed. Superelastic Ni-Ti

* Corresponding author: Lidija Zorko lidija.zorko@uni-mb.si

is highly elastic (rubber-like), while austenitic Ni-Ti is quite strong and hard (similar to titanium). The Ni-Ti material has all these properties, their specific expression depending on the temperature in which it is used [1-3].

The best known properties of Nitinol alloys are super-elasticity and thermal shape memory. An SMA exhibits the shape memory effect (SME) when it is deformed at low temperature and subsequently heated, when it reverts to its original shape. During this process the metal undergoes a complex crystalline-to-solid phase change called martensite-austenite transformation. Sometimes an SMA can exhibit repeated shape changes under no applied mechanical load when subjected to a cyclic thermal load. This behaviour is termed two-way shape memory effect.

Super-elasticity (or pseudo-elasticity) refers to the ability of Ni-Ti to return to its original shape upon unloading after a substantial deformation. The good super-elastic properties of NiTi are based on the stress-induced martensite formation. While many metals exhibit super-elastic effects, only Ni-Ti based alloys appear to be chemically and biologically compatible with the human body.

An important feature of super-elastic Nitinol alloys is that their unloading curves are flat over a wide deflection (strain) range. This allows the design of devices that apply a constant force or load (stress) over a wide range of shapes. The orthodontic arch wire was the first product to use this property.

Nitinol is being used in a wide variety of applications in various fields. Since the initial discovery of Nitinol in 1963, many commercial applications have been developed. During the 1970s, several uses of Ni-Ti in biomedical applications appeared, but it wasn´t until the 1990s that Ni-Ti stents made their commercial breakthrough. By this time, SMAs had found additional applications in air conditioning vents, electronic cable connectors, valves and a variety of other products. Many of the current applications of Nitinol have been in the field of medicine, especially orthodontic arch wires. In this category, Ni-Ti orthodontic arch wires are one of the oldest used materials. Nitinol orthodontic arch wires have been used since the 1970's and are more effective than other alternative materials. In a linear elastic material like stainless steel there is a large amount of force on the tooth for a small amount of corrective motion [1-4].

In the preparation of metallographic samples attention and accuracy are required, as improper handling and processing of samples can lead to unnecessary and unrepaired errors. Special attention is necessary with samples of a typical forms or samples that should be handled more carefully and skilfully [5].

The primary interest of this paper was metallographic preparation of Ni-Ti wires. For this purpose wires from different firms (Forestadent-Germany, Dentaurum-Germany, Ormco-US, GAC International-US) have been used. Most of the wire sizes were with 0.35 mm diameter or some with the size 0.43 mm × 0.63 mm. In this context, the main problem was observation of real martensite or austenite Ni-Ti orthodontic wire microstructure.

Experimental

Ni-Ti wires (had not been previously treated – as received state) were the object of metallographic preparation.

The wires were hot mounted by compound Resin3 and for better integration the clamps were used. Without clamps mounting would be difficult, since Ni-Ti wires are very thin and would not remain in the required position. Also, if the wire is longer than the clamp the wire bends and is no longer suitable for further preparation.

Polishing was performed with a 1μm diamond suspension, 10-15 min at medium tension, on the soft long-napped polishing cloth.

In order to ensure a uniform etching and to obtain realistic and representative microstructure, the ultrasonic cleaning was performed before etching. Otherwise, samples were etched using HF-HNO_3-CH_3COOH, in the ratio 2:5:5, as an etchant **[6]**. First, etching had been performed with the same etchant diluted with water, but the results were not satisfactory. Due to the use of HF, fresh etchant was always required (in our experiments, the application of HF after one hour no longer had the same effect as fresh etchant). Samples were etched for a short period of time (1s). Samples were only dipped in the etchant and then immediately washed under cold running water.

The microstructures of Ni-Ti wires were examined by optical microscope Nikon Epiphot 300.

Results and Discussion

Gaps (Figure 1) occur between the wire and the clamp, because the wire was not well embedded or the mounting compound was not suitable.

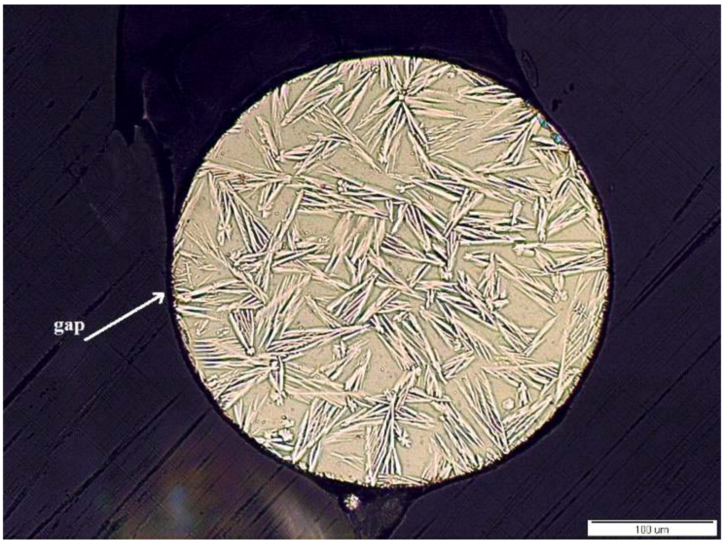

Figure 1. Sample with gap

In order to straighten the investigated side of the sample, the initial stage of grinding was 400 which then continued to the stage 4000. By evenly cut samples, grinding was started at a higher stage (800 or even 1200). After each stage the specimens were examined under the microscope to see if the scratches from the previous level had been removed. It was also necessary to hold the sample carefully and with even pressure, so that the sample was without edge rounding (Figure 2). If that occurred regrinding and re-polishing were required (Figure 3).

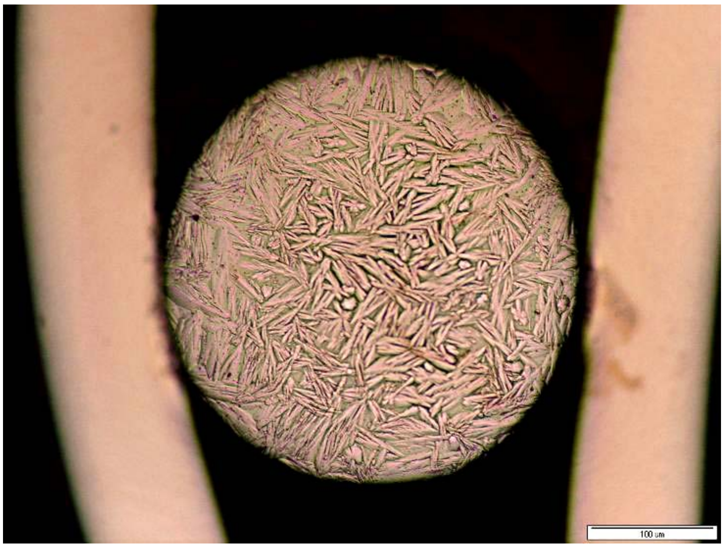

Figure 2. Sample with edge rounding before regrinding and re-polishing

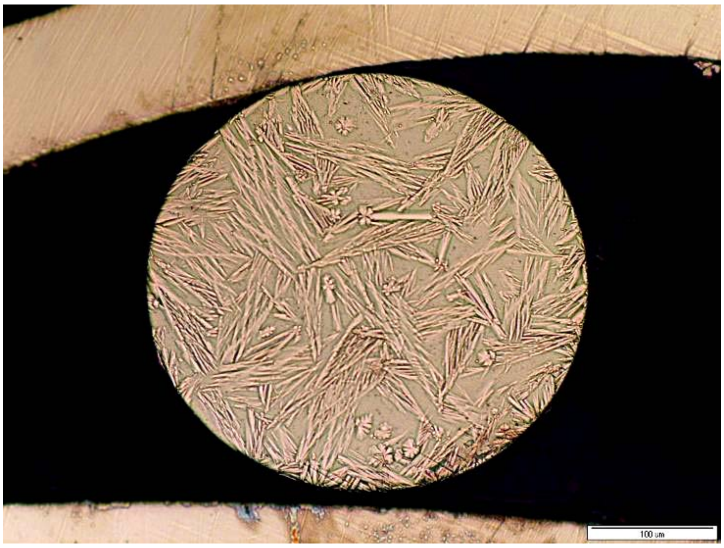

Figure 3. Sample without edge rounding after regrinding and re-polishing

If contamination appeared after polishing (Figure 4) ultrasonic cleaning was necessary (Figure 5). Also, abrasive particles may be pressed into the surface of the sample (Figure 6), probably due to excessive pressure at polishing.

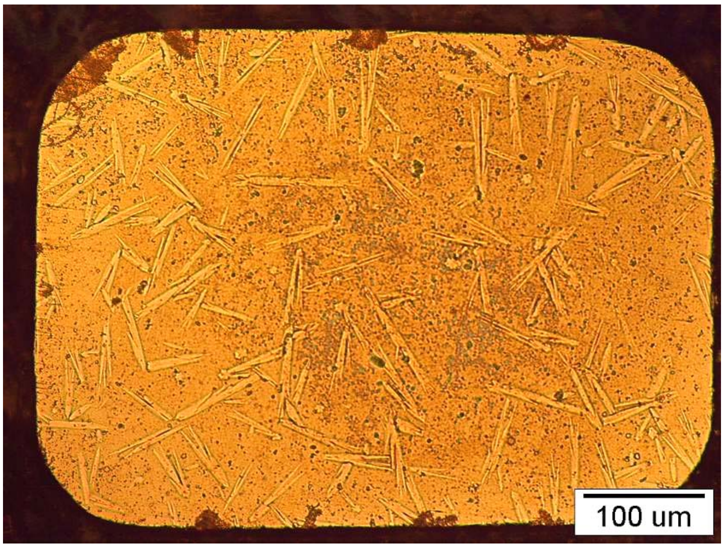

Figure 4. Contaminated sample before ultrasonic cleaning

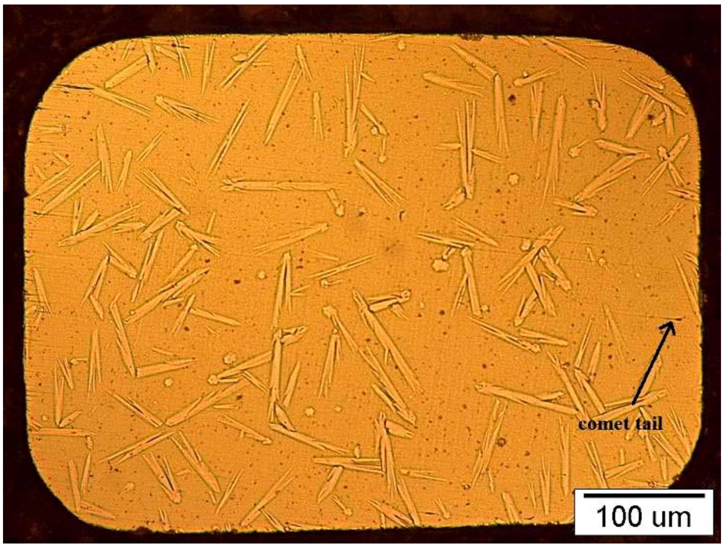

Figure 5. Contaminated sample after ultrasonic cleaning

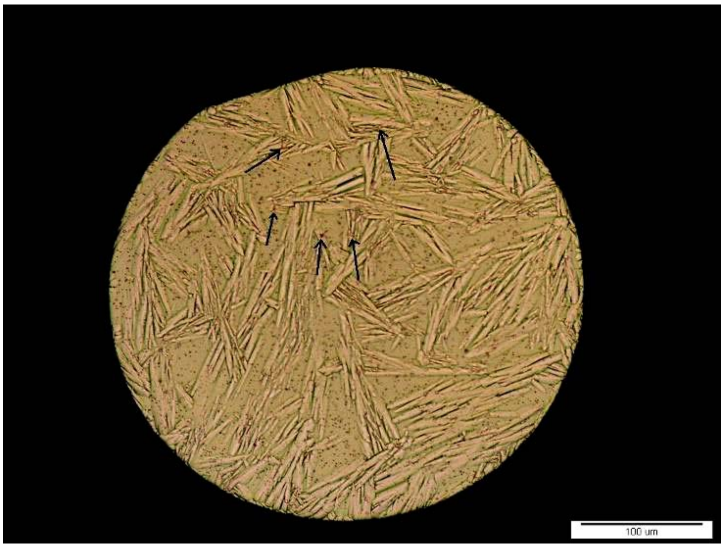

Figure 6. Sample with embedded abrasives

Sometimes scratches are not visible until after etching (Figure 7).

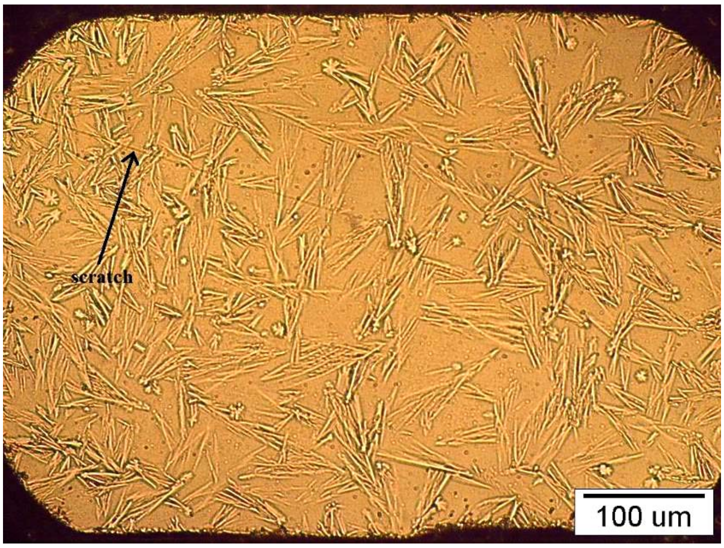

Figure 7. Sample with scratch

Using etchant for a longer period of time resulted in over-etched samples (Figure 8).

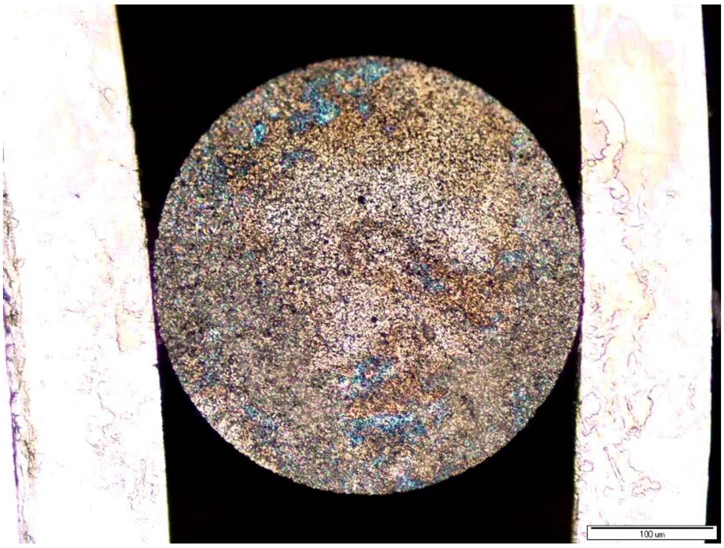

Figure 8. Over-etched sample

In samples with gaps, after etching the etchant bled out during drying and stains occurred on the surface, so ultrasonic cleaning was required (Figure 9).

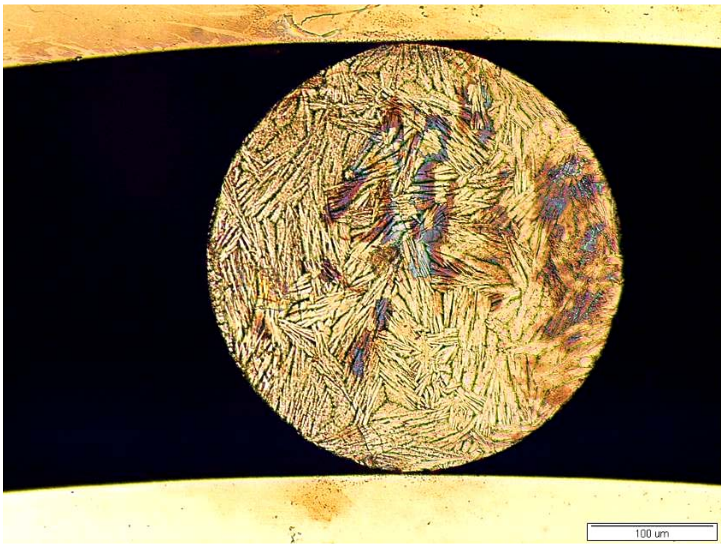

Figure 9. Sample with stain

Conclusion

Metallographic preparation of orthodontic Ni-Ti wires requires selection of the proper mounting compound and use of a clamp. An important factor is to define the initial stage of grinding and, possibly the most crucial factor, is the use of the right polishing cloth and tension (the initial stage of grinding was 400, which were then continued to the stage 4000). Also necessary for quality examination is ultrasonic cleaning. Finally, samples were etched using HF-HNO_3-CH_3COOH, in the ratio 2:5:5 for a short period of time (1s). Consistency with the work, and knowledge of the material and sample preparation procedure itself, may help in the preparation and provision of an ideal model for examination by optical microscope.

Acknowledgments

This paper is part of Bilateral Project SLO/SCG: BI-RS/08-09-003: Biomedical Shape Memory Alloys and EUREKA Programme E!3971 BIO-SMA. The authors thank the Institute of Materials Technology, Faculty of Mechanical Engineering, University of Maribor; University of Ljubljana, Faculty of Natural Sciences and Engineering, School of Dentistry, University of Belgrade Serbia for their support and assistance. The authors also gratefully acknowledge the Ministry of Higher Education, Science and Technology and Slovenian Research Agency and the Ministry of Science and Technological Development of the Republic of Serbia.

References

[1] D. C. Lagoudas: Shape Memory Alloys, Modelling and Engineering Applications, Springer Science + Business Media LLC, New York, 2008

[2] S. Kujala: Biocompatibility and Biomechanical Aspects of Nitinol Shape Memory Metal Implants, Ph.D. Thesis, Oulu 2003

[3] J. Ryhänen: Biocompatibility Evaluation of Nickel-Titanium Shape Memory Metal Alloy, Ph.D. Thesis, Oulu, 1999

[4] D. Stöckel: Nitinol-A Material with Unusual Properties, Endovascular Update, Issue 1, 1998

[5] S. Spaić: Metalografska analiza; Univerza v Ljubljani, FNT, Oddelek za montanistiko, Ljubljana 1993

[6] J. Jafari, S.M. Zebarjad, S.A. Sajjadi: Effect of pre-strain on microstructure of Ni-Ti orthodontic arch wires, Materials Science and Engineering A 473 42-48, 2008