A novel method for sample preparation of SMA spring for micro-structural studies

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Abstract
NiTi alloys are most widely used shape memory alloys (SMA). SMA in spring form is best suited for actuators as they can generate considerable forces and large displacements. Often it is necessary to know the micro structure of SMA to conclude their state as fully austenite or fully martensite at near room temperature. Sample preparation of wire for micro structural study is easier when compared to spring. This paper reveals a novel method of sample preparation for micro structural study using optical and scanning electron microscopes, of commercially available SMA spring in as received condition for which a prior history is unknown.
Keywords: NiTi alloys, actuators, austenite, martensite

1. Introduction
The most widely used shape memory alloys (SMA) are made up of NiTi alloys. Metallographic sample of NiTi SMA spring was prepared for micro-structural study using optical microscope (OM) and scanning electron microscope (SEM)[1,2]. Since the specimen was in spring form, two different ways were tried for mounting the spring in Bakelite inorder to get more polished area for better study.

2. Experimentation
2.1 Microstructure study of NiTi SMA Spring for martensitic temperature
First the spring was kept in vertical condition inside the mount as shown in Figure 1-(a). After mounting, the coil was coming out of the mount one by one with the continuation of polishing. This is shown in Figure 1-(b). Then the spring was kept in near horizontal condition inside the mount as shown in Figure 1-(c). After mounting, the coil was not coming out from the mount while it was polished as shown in Figure 1-(d) and offered enough polished area. Polishing was done in three stages in the following sequence: In first stage, silicon carbide paper
of 1000 grid was used. In second stage, it was polished with 9 micron diamond paste cloth. In third stage, polishing was carried out with 1 micron diamond paste cloth. In the as polished condition OM and SEM images were taken but the microstructure was not visible. Then ultrasonic cleaning was done. But still the microstructure was not visible in OM and SEM images. The etching was done using three different etchants. Firstly Kalling’s reagent [copper chloride-2.5gm, HCL-40ml, methanol-40ml] which is generally used for Ni-based super alloys was used for etching. Secondly Krolls reagent (Nitrlic acid-6ml, HF-3ml, distilled water-100ml) which is generally used for Ti and Ti based alloys was used for etching. Thirdly aquaregia (HCL: HNO3=3:1) which is generally used for stainless steel (Austenite & Ferrite) was used for etching. The microstructure was not seen when all the above three etchants were tried one by one. At last, etchant of HF-HNO3-CH3COOH was used in ratio of 2:5:5 and the microstructure was observed in both OM and SEM and images are shown in Figure 2.
Chemical analysis was also performed using SEM which shows that the SMA spring material was nearly equiatomic: Ti- 49.22% and Ni- 50.78%. The chemical analysis result is shown in Figure 3.

Figure. 2 OM and SEM images

Chemical analysis was also performed using SEM which shows that the SMA spring material was nearly equiatomic: Ti- 49.22% and Ni- 50.78%. The chemical analysis result is shown in Figure 3.
2.2 Microstructure study of NiTi SMA Spring for austenitic temperature

A sample prepared for microstructure study was taken and the top surface of the sample was exposed to hot air produced by the hot air gun to increase the metallic surface of the sample in such a way that it has to reach the temperature greater than the austenite finishing temperature $A_f$ (around 90°C). Then the hot sample was placed under the microscope of the optical microscopy. Although the expectation was to reveal the austenitic microstructure; it showed only a martensitic microstructure. Heating of the metallic sample surface was tried with various setting levels (50% and 100% of its capacity) available in the hot air gun and with different timings (30, 60 and 90 secs). As a result of this experiment, only martensitic structure was shown but the austenitic microstructure was not observed as expected[3,4].

Two different types of optical microscopy were used for observing the microstructure of the sample after heating it by hot air gun. The first microscope was relatively older type as shown in Figure 3(a), the sample was first placed on a wise and elevated to view the microstructure. Due to this delay in placing and positioning of the sample, there is a possibility of heat transfer loss which leads to the reduction in the temperature of the metallic surface of the sample less than or equal to the Martensite finishing temperature $M_f$. The same experiment was repeated with the other new microscope where positioning and placing of the sample does not require more time after heating, due to which there will be less heat loss. But the austenitic microstructure was not observed in the new microscope also. The experimental setups are shown in Figures 4-(a) and 4-(b). The micro structure before heating, just after heating, cooled for 15 minutes after heating, cooled for 25 minutes after heating, cooled completely to room temperature and zoomed view of the metal surface are shown in Figures 4-(c), 4-(d), 4-(e), 4-(f), 4-(g) and 4-(h) respectively. For all conditions it is seen that the martensite microstructure only was visible. The experiment was repeated for four times but the results were same.

During these heating processes, particularly at higher setting level (100% of hot air gun capacity) for 60 and 90 secs, a number of small swellings were observed on top surface of the Bakelite sample mount which are shown in Figures 5-(a) and 5-(b).
2.3 Heat transfer studies with heat flux gauges, thermo-couples and thermography

The austenitic microstructure was not observed in the optical microscopy experiments. So it was decided to know the temperature experienced by the metallic surface of the sample for which three different types of experiments were conducted. They were:

- Exposing the heat flux gauge to hot air gun

**Figure 4** Experimental setup

**Figure 5** Swellings in sample
Exposing the thermocouple to hot air gun
Measuring the temperature of the metallic surface using infrared thermal image

2.3.1 Exposing the heat flux gauge to hot air gun:
During this experiment, hot air was blown on the heat flux gauge by hot air gun with different level settings and different timings and tried to measure the heat flux generated during heating. Initially the heat flux gauge was fixed to a stand and hot air was blown on the gauge by holding the hot air gun in hand as shown in Figure 6-(a). It was difficult to hold the hot air gun in hand for long time due to shaking movements of the hand that caused a variation in the observed values. This was rectified by fixing hot air gun firmly to another stand in such a way that its nozzle end faced the heat flux gauge as shown in Figure 6-(f). The experimental setups are shown in the Figures 6-(c) and 6-(e). The heat flux observed for different setting levels and timings are given in the Table 1.

2.3.2 Exposing the thermocouple to hot air gun:
Similar to previous experiment, here hot air was blown on the thermocouple by hot air gun with different level settings and different timings and the temperature generated was measured during heating [5,6]. The hot air gun was firmly fixed to the stand in such a way that its nozzle end faced the thermocouple as shown in Figure 6-(g). The experimental setups are shown in the Figures 6-(c) & 6-(e). The temperature observed for different setting levels and timings are given in the table 1.

| Setting Level % | Time (secs) | Heat Flux (W/cm²) | Temp (°C) |
|-----------------|-------------|-------------------|-----------|
| 50% (3 pt.)     | 30          | 3.90              | 247       |
| 50% (3 pt.)     | 60          | 3.90              | 252       |
| 50% (3 pt.)     | 90          | 4.16              | 254       |
| 100% (6 pt.)    | 30          | 6.42              | 426       |
| 100% (6 pt.)    | 60          | 9.38              | 461       |
| 100% (6 pt.)    | 90          | 9.38              | 471       |

The temperature display unit, isolation amplifier and heat flux gauge voltage display unit of the experimental setup for both heat flux gauge and thermocouple tests are shown in the Figure 6-(d).
2.3.3 Measuring the temperature of the metallic surface using IR thermal image:

The temperature of the metallic surface of the sample was measured by IR thermal camera after heating it by the hot air gun. The experimental setup for temperature measurement using IR thermal image is shown in Figures 7-(a) & 7-(b). The results are shown in the Figure 8respectively for different timings. After heating the samples, the temperatures of the metallic surfaces are expected to be more being a good thermal conductor unlike bakelite which is an insulator. But it was observed from the IR thermal experimental results that the temperature on the bakelite sample’s top surface was slightly more than the metallic surface temperature at different timings. It was observed that the temperature on both metallic and bakelite surfaces were decreasing with respect to time due to natural convection. The difference in temperature is because of the metallic surface area which is less than 5% of the total top surface area of the bakelite sample. Due to this, the metallic area was heated less by hot air than the bakelite surface area. Also some time is required to place and position the sample under the microscope to see the micro structure which caused some heat loss in the sample[7,8].
3. Conclusions

Knowing the microstructure of SMA to understand their state such as Austenite or Martensite at near room temperature is a prerequisite before starting any experiments on them. Since the specimen is in spring form, two different ways were attempted for mounting the spring in bakelite to get more polished area for clear microstructural study. Initially, the spring was kept in vertical condition inside the mount but during polishing, the coil was coming out of the mount one by one. Then the spring was kept in horizontal condition inside the mount in which the coil was not coming out. A novel method is proposed to prepare a metallographic sample of NiTi SMA spring for microstructural studies. Different combinations of etchants have been tried out on the polished surfaces of metallographic samples and finalised the appropriate proportions.
of etchants. Microstructural studies have been carried out at both martensitic and austenitic temperatures using optical microscope (OM) and scanning electron microscope (SEM). Chemical analyses also performed using the same metallographic sample. Martensitic microstructure was observed in both OM and SEM at near room temperature. But at Austenitic temperature, it was not possible to observe austenitic microstructure from the sample. This could be due to the fact that the metallic area was heated less by hot air than the bakelite surface area.

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