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Research Article

AGING EFFECTS ON TRANSFORMATION TEMPERATURES AND ENTHALPIES FOR TiNi ALLOY

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ABSTRACT
Shape memory alloys are frequently used today. For this reason, researches are more focused on this subject. In this study, transformation temperature and enthalpy changes caused by aging of TiNi alloy were investigated from thermally induced shape memory alloys. The results obtained from the experiments performed in 21 different aging environments were evaluated by using graphs. The results showed us how effective the variable parameters used in the experiments are on the alloy.

Keywords: *Aging Effect, Shape Memory Alloy, TiNi, DSC, Martensitic Transformation*

1. INTRODUCTION

Nitinol, discovered in the early 1960s, started a new material concept. The superior capabilities of shape memory alloys inspired many applications. This alloy, which has been subject to many applications from the day it was discovered to the present day, has progressed rapidly.

Many areas are used from health to space technology (Birman 1997; Van Humbeeck, 2001; Lagoudas, 2008; Yamauchi *et al.* 2011; Jani *et al.* 2014). The expression showing usage areas according to years is presented in Fig. 1.

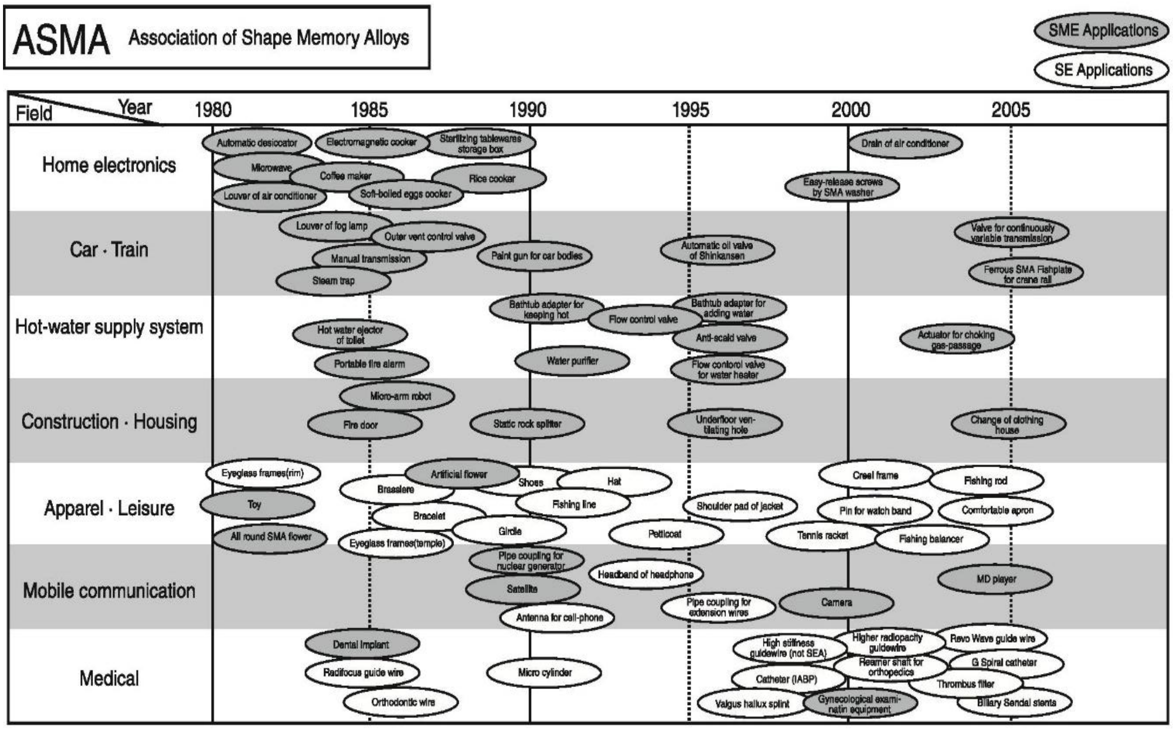


Figure 1. History of SMA application in Japan (Yamauchi, Ohkata, Tsuchiya and Miyazaki 2011)

After the discovery of shape memory alloys, many alloy combinations have been tried. But the most functional of these is the TiNi alloy. It is used very frequently in the health field, especially since it performs very well in biocompatibility. At the same time, it has the attractiveness of having transformation temperatures close to room temperatures (Shabalovskaya, 1996; Es-Souni *et al.* 2005). Shape memory alloys can be induced by many different methods. However, in industrial applications, temperature-inducing alloys are generally used such as thermostat (Wayman, 1993).

Therefore, the alloys used are exposed to heat afterwards. As this cycle continues, an aging occurs on the material. This aging causes changes in the character of the material in the process. This change in material is important in product design, product life and quality (Stachowiak *et al.* 1988; Otubo *et al.* 2008; Frenzel *et al.* 2010; Canbay *et al.* 2017).

In this study, experiments were carried out by aging the commercial TiNi alloy at different aging temperatures and waiting times. The results of the maximum temperature and enthalpy values of the austenite phases of the alloys were obtained with a differential scanning calorimeter (DSC) instrument. The values are then reviewed modeled thanks to 3D graphics.

2. EXPERIMENTAL

In our experimental work we used a commercial product, a 0.7 mm diameter TiNi alloy. We split this alloy into 21 equal parts and made it ready for use in experiments. The chemical composition of the material was obtained by energy dispersive X-ray micro analyzer and the results are shown in Table 1.

Table 1. Specimen chemical composition results

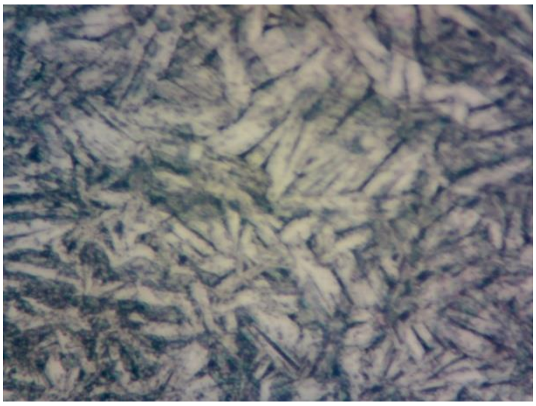
Element	Weight%	Atomic%
Ti	50.62	55.68
Ni	49.38	44.32

In aging experiments, 7 different waiting times were selected at 3 different temperatures. The products obtained by performing 21 tests in total were collected the conversion temperatures and enthalpy values in the DSC device. The results obtained with the parameters of the experiments performed are shown in Table 2.

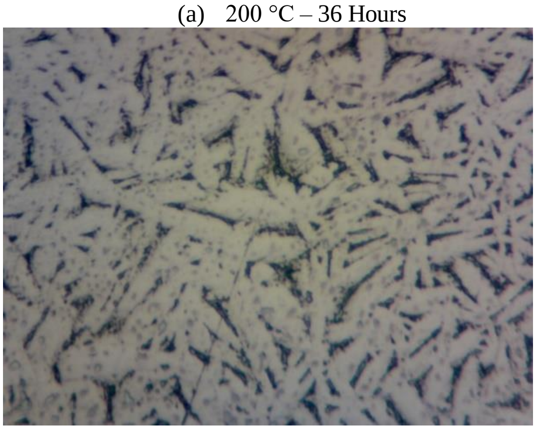
Table 2. Aged TiNi samples martensite transformation analyze results

Item	A_{max} (C°)	$\Delta H_{M\rightarrow A}$ (J.g^{-1})	M_{max} (C°)	$\Delta H_{A\rightarrow M}$ (J.g^{-1})
200 C° - 0.5h	47.12	-5.43	40.45	3.21
200 C° - 1h	47.08	-3.96	46.94	1.49
200 C° - 3h	46.84	-3.79	40.93	1.74
200 C° - 7h	47.43	-4.40	41.59	2.09
200 C° - 24h	47.08	-3.45	41.01	2.23
200 C° - 36h	47.55	-6.30	41.08	3.11
200 C° - 48h	48.05	-3.81	39.14	1.93
300 C° - 0.5h	47.92	-3.41	43.03	1.32
300 C° - 1h	49.94	-4.49	43.01	2.31
300 C° - 3h	47.59	-5.04	40.73	2.73
300 C° - 7h	53.69	-4.92	47.18	1.9
300 C° - 24h	53.59	-6.24	46.5	3.75
300 C° - 36h	53.97	-4.53	47.51	1.72
300 C° - 48h	54.58	-5.55	47.68	3.71
400 C° - 0.5h	51.25	-4.36	44.46	2.86
400 C° - 1h	51.35	-3.57	44.35	3.2
400 C° - 3h	51.36	-4.09	45.45	2.72
400 C° - 7h	51.8	-4.36	44.59	3.2
400 C° - 24h	51.72	-5.25	44.07	3.98
400 C° - 36h	52.88	-5.58	45.17	4.63
400 C° - 48h	53.97	-5.96	45.81	4.29

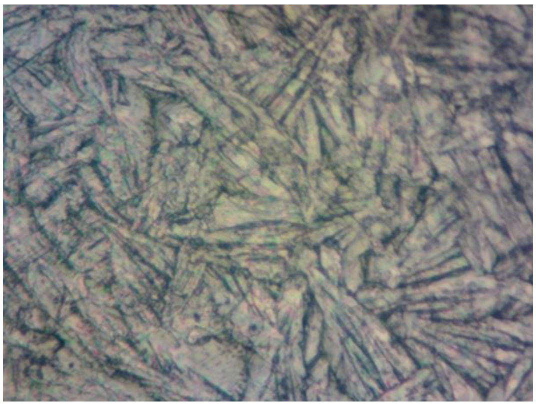
Furthermore, the specimens were applied fine polishing and etched with 10%HF-60%HNO_3-30%CH_3COOH solution. Microstructure observations of alloys by optical microscopy are depicted in Fig. 2.



(a) 200 °C – 36 Hours



(d) 400 °C – 36 Hours



(b) 300 °C – 36 Hours

Fig. 2. The optic micrograph images TiNi Alloys

Fig. 2 shows images of the optical microscope at different temperatures but at the same dwell times. The martensite plates with different orientations in the images are easily observed.

3. DISCUSSION

The results are created as a contour plot using the graphics program. The obtained graphs show the relationship between Amax, Mmax and enthalpy values results with temperature and waiting times. Austenite maximum temperature and martensite maximum temperature values are the tops of the phase change peaks in the DSC graph. The area under the curve is the enthalpy value that represents the amount of energy required during solid state phase transformation and that's shown in Fig. 3.

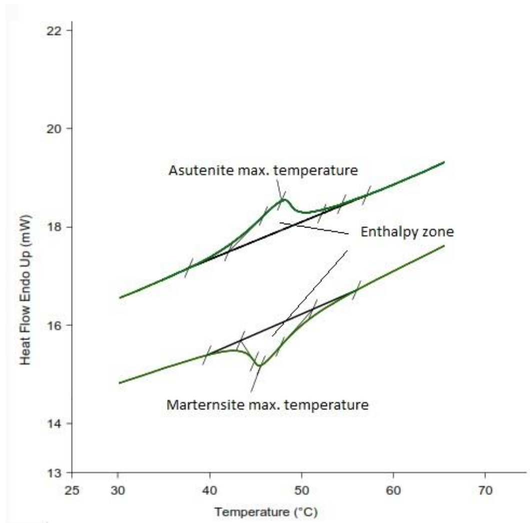


Fig. 3. A sample DSC plot

These values are endothermic and exothermic. This is why the enthalpy values in some of the results obtained are negative. In our study, the 2D contour plots and dynamic models were generated by sigma plot software using the experimental results. The graphs are shown in Fig. 4.

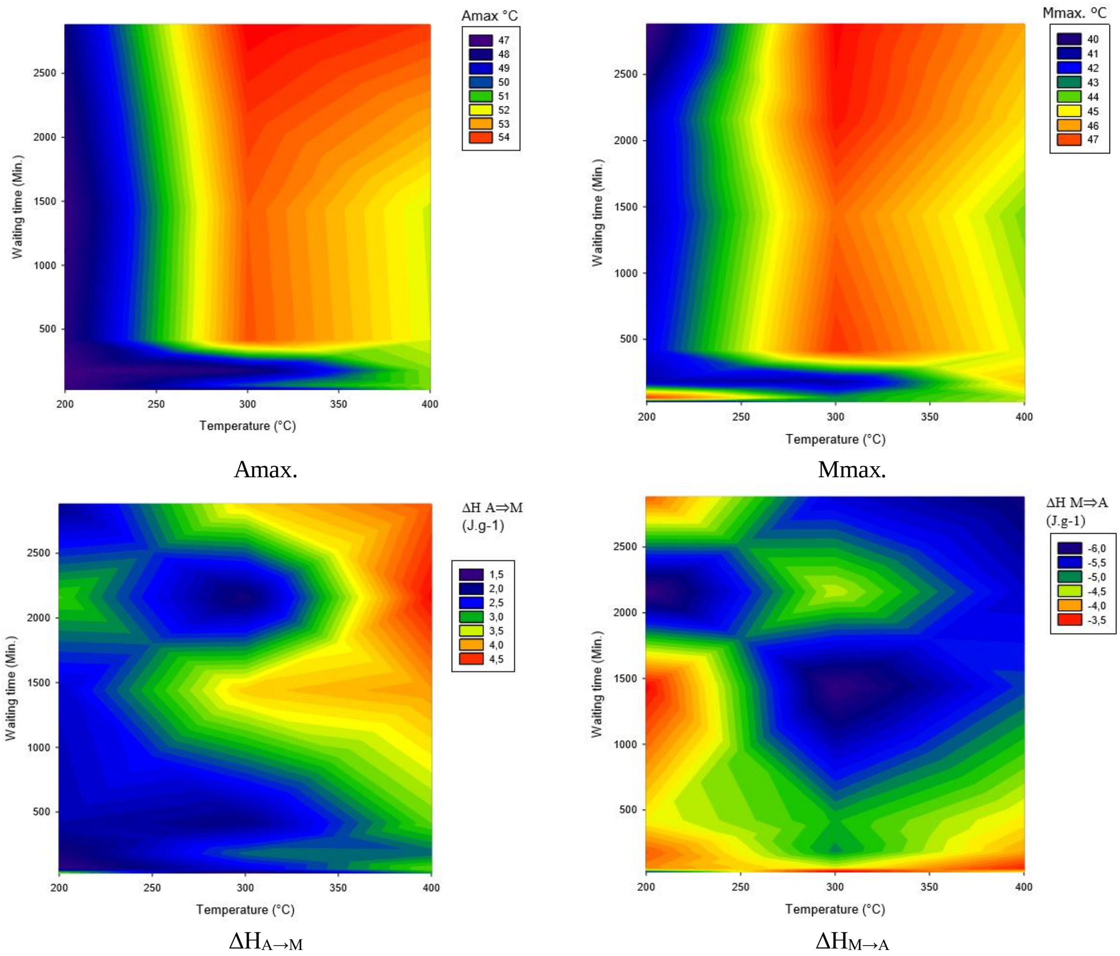


Fig. 4. The contour plots of martensite and austenite phase transformation temperatures and enthalpies

Also, the nonlinear regression - dynamic fitting results are depicted following equations.

$$Amax=25.8559+0.1398*(AT)+0.0017*(WT)-0.0002*(AT)^2+-2{,}4(10^{-7})(WT)^2 \quad (1)$$

$$Mmax=-23.3457-0.1287*(AT)-0.0009*(WT)-0.0006*(AT)^2+5.79(10^{-7})(WT)^2 \quad (2)$$

$$\Delta H_{A\rightarrow M}=-4.2743-0.0551*(AT)-0.0008*(WT)-4.15(10^{-5})*(AT)^2-4.77(10^{-7})(WT)^2 \quad (3)$$

$$\Delta H_{M\rightarrow A}=-5.5329-0.0564*(AT)-0.0030*(WT)-2.89(10^{-5})*(AT)^2+2{,}43(10^{-7})(WT)^2 \quad (4)$$

In Fig. 4, the Amax. And Mmax. a smooth transition was observed along the temperature axis. These transitions in the opposite direction is decreased after 300 degrees. The greatest change in temperature deviations was seen at 300 degrees relative to these graphs. However, an irregularity in the transit at the horizontal line corresponding to 60 minutes is clearly visible. The defection in this section is caused by residual stresses of the alloy. It was seen that the stresses on the material did not cause any trouble during the high waiting periods. When the enthalpy graphs are examined, it is determined that there are different soft passages but there are regional distortions and irregularities at 300 degrees. The colour ranges on the enthalpy charts are different because they are endothermic and exothermic reactions.

4. RESULTS

It is very important to know the character of the material when designing the product. The changes in the aging process of TiNi alloys, which are in the class of smart materials and have the most use, have been investigated. In our study, Ti55.68Ni44.32 (at.%) alloys were tested at 7 waiting times at 3 different temperature values. The austenite and martensite max temperature values determined by the results obtained by the DSC device and graphs of enthalpy changes were generated. As a result of the graphics, an increase of up to 300 degrees was found to be followed by a decrease in martensite and austenite maximum temperatures. These transitions cannot be observed due to waste stresses around 60 degrees. Enthalpy values have not been found in a regular transition but regional variations have occurred at 300 degrees.

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