Formability of Annealed Ni-Ti Shape Memory Alloy Sheet

K J Fann¹, J Y Su¹ and C H Chang¹

¹ Department of Mechanical Engineering, National Chung Hsing University, Taichung 40227, Taiwan

kjfann@nchu.edu.tw

Abstract. Ni-Ti shape memory alloy has two specific properties, superelasticity and shape memory effect, and thus is widely applied in diverse industries. To extend its application, this study attempts to investigate the strength and cold formability of its sheet blank, which is annealed at various temperatures, by hardness test and by Erichsen-like cupping test. As a result, the higher the annealing temperature, the lower the hardness, the lower the maximum punch load as the sheet blank fractured, and the lower the Erichsen-like index or the lower the formability. In general, the Ni-Ti sheet after annealing has an Erichsen-like index between 8 mm and 9 mm. This study has also confirmed via DSC that the Ni-Ti shape memory alloy possesses the austenitic phase and shows the superelasticity at room temperature.

1. Introduction

Recently Ni-Ti shape memory alloy has been broadly used as biomedical material [1] not only due to its specific shape memory effect and superelasticity [2] but also because of good compatibility and well corrosion resistance in human bodies, such as coronary or vascular tents [3], bone clamps [4], dental braces [5], and so on for medical devices [6]. In the meantime, there are a variety of industrial applications implemented with Ni-Ti shape memory alloy [1] such as dampers, sensors, actuators, aligning pins, tube fittings and disc springs, and so on [7], by means of the particular properties of free recovery and constrained recovery based on shape memory effect.

The above mentioned shape memory effect is actually caused by phase transformation between austenite at high temperature and martensite at low temperature [8]. The shape formed in austenite at high temperature will be recovered, if the part is deformed in martensite at low temperature and the load is released, by raising the temperature, at which the austenite is stable. This shape recovery is called as free recovery. The superelasticity is in fact rubber-like behaviour, which is caused as well by phase transformation between austenite and martensite at high temperature where the austenite is table [9].

The parts made for the above mentioned applications are from Ni-Ti shape memory alloy tube or wire, mainly due to the material not having good machining properties, so that it must be formed into a parts. However, most of Ni-Ti shape memory alloy shows its superelasticity at room temperature and most of formed shape will spring back after forming. Therefore the forming process must be done in thermomechanical manner [10], in that Ni-Ti shape memory alloy part is formed in hot state, in which the ill-favored superelasticity will not present. On the other hand, such hot forming leads to limit the part shape only formed by tube or wire, not by sheet, which are usually formed at room temperature because of their geometrical characteristics to easily dissipate the heat. Furthermore, to secure or to memorize the shape of Ni-Ti shape memory alloy part, it needs further precipitation treatment at high temperature. Thus the tool sets used in the forming process are needed to clamp the parts to keep their
shapes during the shape memory treatment at high temperature, which is a cost intensive process and needs lots of investment for the tool sets [11]. To overcome both above mentioned hindrances, Fann and Huang [12] therefore proposed a cold forming process, in that the shape memory alloys are first solid solutionized, then formed at room temperature, and thereafter aged for shape memory again at high temperature. Even this process would lose part of the shape formed at room temperature during the precipitation treatment, it is still worth to use it to form the shape memory alloy parts for saving the cost of the tool sets. However, Fann and Su [13] shows the solid solutionized Ni-Ti shape memory alloy sheet shows relatively low formability. Therefore this article is aimed to investigate the formability of Ni-Ti shape memory alloy sheets at room temperature, which are annealed at different temperatures [14].

2. Methods and Setup

2.1. Material

The material acquired for this study was a commercial Ni-rich (50.0-50.8at% Ni) Ni-Ti shape memory alloy sheet having a thickness of 0.9 mm. The chemical compositions of the alloy sheet was determined by a glow discharge spectrometer as shown in Table 1 and had 50.67at% Ni and 49.24at% Ti after converting weight percent (wt%) to atomic percent (at%). The content of Fe is as little as 0.09at% and can be regarded as a minimal contaminant during casting, so that the material can be considered as a pure binary Ni-Ti system.

|       | Ni   | Ti   | Fe   |
|-------|------|------|------|
|       | 55.73| 44.17| 0.10 |

2.1.1. Determination of Transformation Temperatures. The characteristic temperatures of phase transformations occurring in the Ni-Ti shape memory alloy sheets were determined by a Differential Scanning Calorimetry (DSC) according to ASTM standard F2004 [15]. Samples were prepared by cutting them from the sheet blank, descaled with hydrofluoric-sulfuric acid and had a weight between 25 and 45 mg. By cooling the samples from 150°C to -150°C and heating from -150°C to 150°C with a rate of 10±0.5°C/min, the heat flow rate between the samples and a reference of empty aluminum pan was recorded. The characteristic transformation temperatures are then determined by drawing the tangents on the heating and cooling spikes from the DSC plot.

2.1.2. Annealing. The Ni-Ti shape memory alloy sheet was cut into a circular shape with a diameter of 36 mm by waterjet. Then the circular specimen was placed in a heated chamber at 300°C, 350°C, 400°C, 550°C, 600°C, and 650°C, respectively, for one hour and subsequently cooled in the still air at laboratory environment. Figure 1 shows the specimen having different colors in appearance after annealing.

2.1.3. Hardness Test. After annealing the mechanical property of the Ni-Ti shape memory alloy sheet blanks were determined by Rockwell hardness test according to ASTM standard D18 [16]. The average was taken from the Rockwell hardness measured at the mostly wide separated four points on each alloy sheet blank.
2.2. Erichsen-Like Cupping Test
Before executing any cold forming experiments of the Ni-Ti shape memory alloy sheets at room temperature, an Erichsen-like cupping test should be done to determine their process parameter. The Erichsen-like cupping test was actually served as a pre-test for preparing the tools of experiments by seeing how deep the Ni-Ti shape memory alloy sheet could be formed. Because the dimension for the cupping test deviated a little bit from the standard Erichsen cupping test [17], it was called in this study as Erichsen-like cupping test. Besides the punch diameter, the outer diameter of the blank was different to the standard as well. Figure 2 shows the schematic experiment arrangement used in the study, which was conducted on a computer universal testing machine. The sheet blank was clamped between the blankholder and the die by four M12 bolts with a fastening torque in 5 N·m, which induced a larger clamping force other than the standard. Subsequently a hemispherical punch with a diameter of 30 mm descended with a traveling speed of 10 mm/min through the hole of the blankholder on the blank as well as into the cavity of the bottom die. Once the sheet blank could not endure the punch load and fractured, the punch stroke would be recorded as Erichsen-like index, which indicates the formability of the Ni-Ti shape memory alloy sheet.

![Figure 2. Schematic illustration of Erichsen-like cupping test](image)

3. Results and Discussion

3.1. Transformation Temperatures
Figure 3 shows the DSC curve of the Ni-Ti shape memory alloy as received. On the upper part of the DSC curve, it can be seen that the martensitic start temperature (\(M_s\)) is about -53°C at the beginning of the spike, and martensitic finish temperature (\(M_f\)) should be lower than -150°C at the transition after the spike drops. On the bottom part of the DSC curve, the austenitic start temperature (\(A_s\)) is found about -47°C and the austenitic finish temperature (\(A_f\)) is then about -19°C. This DSC curve shows that the Ni-Ti shape memory alloy formed at room temperature, which is higher than \(A_f\), is possessing the austenitic phase and showing the superelasticity.

![Figure 3. Transformation temperatures - martensitic start (\(M_s\)), austenitic start (\(A_s\)), and austenitic finish (\(A_f\)) obtained for the Ni-Ti shape memory alloy as received.](image)
3.2. Rockwell Hardness
Figure 4 shows the average HRC readings obtained from the Rockwell hardness tests on each Ni-Ti shape memory alloy sheet blanks annealed at different temperatures. It can be seen that the higher the annealing temperature, the lower the HRC value, even the hardness shown at 550°C departs from the trendline. With such high HRC values, the Ni-Ti shape memory alloy sheet shows very high strength and hard to deform.

![Figure 4](image)

**Figure 4.** Rockwell hardness obtained on the Ni-Ti shape memory alloy annealed at different temperatures.

3.3. Erichsen-Like Cupping Test
Figure 5 shows the fractured specimen obtained from the Erichsen-like cupping test to each Ni-Ti shape memory alloy sheet blanks annealed at different temperatures. Most of them have the fractured location around the corner radius of the bottom die, while one of them has the fracture occurring at the final contact region of the punch to the sheet. The corner radius of the bottom die, which is not defined in the standard, is 1 mm in this study, which might cause the failure occurring there.

![Figure 5](image)

**Figure 5.** Fractured specimen after Erichsen-like cupping tests of the Ni-Ti shape memory alloy annealed at different temperatures.

Figure 6 shows the punch stroke-load curves obtained from the Erichsen-like cupping test to each Ni-Ti shape memory alloy sheet blanks annealed at different temperatures until the sheet blanks fractured. Even the HRC on the sheet annealed at 550°C higher than the trendline as shown in Figure 4, its maximum punch load still locates on the reasonable trail. As expected, the higher the annealing temperature, the lower the maximum punch load. However the Erichsen-like index shown in Figure 6, where the punch load dropped, has different results, in which the higher the annealing temperature, the lower the index or the lower the formability, even the indices obtained from intermediate temperatures fluctuate slightly from the trendline. In general, the Erichsen-like index has between 8 mm and 9 mm.

4. Conclusions
This study has accomplished the hardness test and Erichsen-like cupping test at room temperature to investigate the formability of the Ni-rich Ni-Ti shape memory alloy sheet annealed at 300°C, 350°C, 400°C, 550°C, 600°C, and 650°C, respectively, for one hour and subsequently cooled in the still air.
As a result, the higher the annealing temperature, the lower the hardness and the lower the maximum punch load obtained from the Erichsen-like cupping test as the sheet blank fractured. However the Erichsen-like index shows that the higher the annealing temperature, the lower the index or the lower the formability. In general, the Ni-rich Ni-Ti shape memory alloy sheet after annealing has an Erichsen-like index between 8 mm and 9 mm.

This study has also confirmed by means of DSC that the Ni-Ti shape memory alloy acquired in this study possesses the austenitic phase and shows the superelasticity at room temperature, which causes significant spring back if it is formed cold.

![Figure 6](image)

**Figure 6.** Punch stroke-load curves obtained from the Erichsen-like cupping tests to the Ni-Ti shape memory alloy annealed at different temperatures.

5. References

[1] Wayman CM 1980 *JOM* **32** 129
[2] Shabalovskaya SA 1995 *ICOMAT* 1199
[3] Sigwart U 1996 *Endoluminal Stenting* (London: W. B. Saunders)
[4] Kim DJ, Eun JP and Park JS 2012 *J. Korean Neurosurg. Soc.* **52** 21
[5] Duerig TW, Pelton AR and Stöckel D 1996 *Metall* **50** 569
[6] Duerig T, Pelton A and Stöckel D 1999 *Mater. Sci. Eng. A* **273–275** 149
[7] Hodgson DE, Wu MH and Biermann RJ 1990 *ASM Handbook, Volume 2: Properties and Selection: Nonferrous Alloys and Special-Purpose Materials* ASM Handbook Committee (Materials Park: ASM International) 897
[8] Chang LC and Read TA 1951 *T. Metall. Soc. AIME J. Metals* **189** 47
[9] Christian JW 1965 *The Theory of Transformations in Metals and Alloys* ed. Christian JW (Oxford: Elsevier) 1102
[10] Wayman CW and Duerig TW 1990 *Engineering Aspects of Shape Memory Alloys* ed. Duerig TW, Melton KN, Stöckel D and Wayman CM (London: Butterworth-Heinemann) 3
[11] Fann KJ and Hsu HC 2014 *Adv. Mater. Res.* **939** 430
[12] Fann KJ and Huang PM 2015 *Key Eng. Mater.* **661** 98
[13] Fann KJ and Su JY 2017 *Proc. Inter. Conf. Mater. Mech. Manuf. Eng. (ICMMME)*
[14] Huang X and Liu Y 2001 *Scripta Mater.* **45** 153
[15] American Society for Testing and Materials 2010 *Standard Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis* (West Conshohocken: ASTM International) Designation: F2004-10.
[16] American Society for Testing and Materials 2017 *Standard Test Method for Rockwell Hardness of Metallic Materials* (West Conshohocken: ASTM International) Designation: E18-17.
[17] International Organization for Standardization 2013 *Metallic materials - Sheet and strip - Erichsen cupping test* ISO 20482:2013(E)

Acknowledgments

This research was financially supported by the Ministry of Science and Technology of the Republic of China with the grant coded MOST 105-2221-E-005-029.