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ScienceDirect

Acta Materialia 89 (2015) 298–304

Acta MATERIALIA

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Martensite aging – Avenue to new high temperature shape memory alloys

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Received 8 January 2015; accepted 16 January 2015

Abstract—High-temperature shape memory alloys are attractive for efficient solid state actuation. A key criterion for shape memory alloys is the martensite start temperature. The current study introduces a concept for increasing this temperature of alloys initially not suited for high-temperature actuation. Aging of stress-induced martensite, referred to as SIM-aging in the current work, is able to increase the martensite start temperature by about 130 °C as demonstrated in the present study for a Co–Ni–Ga shape memory alloy. The increase of transformation temperatures can be explained based on the concept of symmetry-conforming short-range order. Following SIM-aging the Co–Ni–Ga alloy shows cyclic actuation stability at elevated temperatures. While martensite aging has always been viewed as detrimental in the past, it can actually be exploited to design new classes of high-temperature shape memory alloys with excellent properties.
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Keywords: Shape memory; Pseudoelasticity; Actuation; Microstructure; Functional stability

1. Introduction

Advances in materials are a crucial factor for technological progress as they are of paramount importance for the development of energy and resource efficient processes. Functional materials can provide high power output and are the key for smart applications using sensors and actuators. Shape memory alloys (SMAs) represent a class of smart materials that can deliver large reversible strains (≈10%), and thus, are of interest for actuation in numerous fields [1–5]. The most widely employed SMA so far is nickel–titanium (Ni–Ti) [1,2,6,7]. Unfortunately, Ni–Ti SMAs can only be used up to temperatures of about 80 °C [3]. An increase of the martensite start temperature (M_s) of the SMAs allows for extending their application range to higher temperatures [3]. Ternary Ni–Ti–X alloys have been designed which contain high amounts of noble or refractory elements (X) [3,8–10]. The former turned out to be too expensive for bulk application. Recently, β-titanium alloys have been proposed as a promising alternative [11–13]. The binary Ti–Ta system shows M_s temperatures well suited for application at elevated temperatures, but suffers from microstructural instability, which in turn leads to functional degradation [11,13].

Another issue hindering robust application is the stabilization of the martensitic phase [14–17] found in many shape memory alloys. As martensite stabilization imposed by aging phenomena leads to a shift of transformation temperatures, a stable actuation response in a well-defined temperature range, crucially needed for application, cannot be obtained in many SMAs. In order to determine the mechanisms responsible for martensite aging, numerous studies have been conducted over the last decades [14–20]. A variety of microstructural mechanisms have been identified contributing to martensite stabilization. Detwinning of martensite leading to a decrease of internal stresses supporting the reverse transformation and concurrently the necessity of forming new habit planes is one important degradation effect [18]. Another mechanism appears to be pinning of moving interfaces by defects ("kinetic" stabilization), which is associated with slow heating rates [19]. Most importantly, re-ordering mechanisms, termed "static" stabilization [19], have been identified to govern martensite stabilization. Depending on alloy thermodynamics, a change in long-range or short-range order can take place. As diffusion is a prerequisite for these re-ordering mechanisms, the density of defects, *i.e.* vacancies and anti-site defects, is an important factor [15–19]. Otsuka and Ren

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http://dx.doi.org/10.1016/j.actamat.2015.01.042
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have introduced the concept of symmetry-conforming short-range order (SC–SRO), which is able to explain the martensite aging phenomenon as well as rubber-like behavior (RLB) and microstructure memory effect in SMAs [14,15]. They showed that martensite and austenite can be stabilized by decreasing their free energy resulting from symmetry changes through transformation and diffusion of defects within sub-lattices [14,15]. However, not all SMAs are prone to martensite stabilization [15]. As an indicator for the susceptibility of SMAs to martensite stabilization, the reduced martensitic transformation temperature M_s/T_m has been proposed, where T_m is the melting point of the alloy [15]. For $M_s/T_m < 0.2$, the aging time for RLB tends to infinity, and martensite stabilization does not pose a problem, *e.g.* in Ni-Ti at room temperature. For $M_s/T_m > 0.2$ aging kinetics start to increase rapidly, and thus, effects of aging manifest themselves almost instantaneously in Cu-based SMA [15].

The current study aims at introducing a new class of high-temperature shape memory alloys (HT-SMAs). The ideal SMA actuator for elevated temperature applications should feature large displacements in a small volume, *i.e.* the strains linked to the phase transformation in the SMA should be as high as possible. Moreover, M_s should be easy to adjust, expensive elements should be avoided and the material should feature high ductility for ease of processing as well as a high melting point in order to withstand undesirable aging effects. Based on these considerations Co–Ni–Ga has been chosen as an appropriate alloy.

2. Experimental details

Large ⟨001⟩-oriented single crystals of a $Co_{49}Ni_{21}Ga_{30}$ alloy grown by the Bridgman technique under a helium atmosphere were employed in the current study. Cuboidal samples with the dimensions 4 mm × 4 mm × 8 mm were cut from the single crystal by electro-discharge machining (EDM) such that the loading axes coincided with the ⟨001⟩ direction. Following EDM, the samples were mechanically ground in order to remove any residue from processing. For solutionizing for 12 h at 1200 °C, the samples were encapsulated in evacuated quartz glass tubes. Subsequently, they were subjected to the different aging heat treatments. Two sets of the solutionized Co–Ni–Ga samples were aged in the load-free condition in the austenitic state, for 8.5 h at 300 °C and for 20 min at 400 °C, respectively. Aging treatments with superimposed compressive loads were conducted employing the same temperatures and times. For aging under load, a servo-hydraulic testing rig equipped with a high-temperature extensometer was employed. Heating was conducted using controlled convection furnaces. Temperatures were measured by a thermocouple attached to one side of the sample. The aging experiments were performed in strain control. Thus, different strain levels could be chosen that allowed for establishing different fractions of stress-induced martensite. Loading was always imposed after initial heating of the sample. Following aging in the (partially) martensitic state (referred to as SIM-aging in the current work), the specimens were then tested under different loading conditions. The pseudoelastic (PE) material response was characterized employing the same setup as used for SIM-aging. Tests were done at 50 °C in strain control and were stopped at a maximum load level of −1200 MPa in order to avoid gross plastic deformation of the martensite. Subsequently, the specimens were unloaded in force control. Strain–temperature tests were conducted at different superimposed constant compressive stress levels ranging from −100 MPa to −300 MPa. Heating/cooling rates of 2.5 °C s^{-1} were employed in the present study (tests at temperatures above RT). For tests below RT a nitrogen heating/cooling system was employed at heating/cooling rates of 0.1 °C s^{-1}. Thermal cycling was conducted up to 30 cycles in order to assess the stability of the strain–temperature response. For microstructure analyses a transmission electron microscope (TEM) operating at a nominal voltage of 200 kV was used. For TEM work, small discs were cut normal to the loading direction from the crystals. These discs were mechanically ground down to 0.15 μm followed by conventional twin-jet polishing using a perchloric acid solution containing 60 ml perchloric acid, 340 ml butanol and 600 ml methanol. Alternatively, focused ion beam TEM foil micromachining was performed in a FEI Quanta 200 3D.

3. Results and discussion

For the current study, ⟨001⟩-oriented single crystalline $Co_{49}Ni_{21}Ga_{30}$ was selected in order to reveal the full potential of the SIM-aging procedure. Work on polycrystalline material is currently in progress and will be subject of future work. Initially developed for magnetic shape memory applications [21], studies in recent years have revealed its high-temperature pseudoelastic properties [22–24]. A very weak dependence of stress on temperature, *i.e.* a small Clausius–Clapeyron (CC) constant, was found for Co–Ni–Ga [22]. In addition, it has been demonstrated that this material can yield maximum strains of about 5% in compression and 8% in tension [23,24]. However, due to its low M_s of about −5 °C (without superimposed stress) [23], Co–Ni–Ga has only been considered as an elevated temperature pseudoelastic material, but not as a HT-SMA for actuator applications. Featuring a T_m of about 1350 °C, its M_s/T_m is equal to 0.17 indicating its stability against martensite aging at ambient temperatures. The current work will introduce an approach referred to as SIM-aging that enables controlled martensite aging of Co–Ni–Ga. Following SIM-aging, the material is no longer pseudoelastic in the temperature range considered and can be used for HT-SMA actuator applications.

3.1. The effect of aging on the pseudoelastic response and microstructure evolution

Fig. 1 depicts pseudoelastic hysteresis curves from three ⟨001⟩-oriented single crystals tested in the solutionized condition. The initial cycle is plotted for each specimen (solid line on the bottom) to reveal that the initial stress–strain response of all samples tested is similar and that a fully austenitic initial condition was established for each sample. The minor differences seen are due to specimen to specimen variations attributed to the susceptibility of the stress level to small changes in chemical composition. This initial test of the material response was conducted on each sample. Following both aging treatments (8.5 h at 300 °C

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