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A new experimental method for measuring stress-temperature phase diagram in shape memory alloys

Emil Bronstein, Eilon Faran *, Doron Shilo

Department of Mechanical Engineering, Technion, Haifa 3200003, Israel

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ABSTRACT

The martensitic phase transformation in shape memory alloys (SMA) can be described by stress-temperature phase diagram, which provides fundamental scientific and applications-related information on this important process. We present a new experimental method that enables direct measurement of the complete stress-temperature phase diagram of SMA within a single experiment. The experimental setup is placed under an optical microscope and allows complementary visualization of the microstructure evolution throughout the phase transformation. The method is demonstrated on a SMA Ni-Mn-Ga single crystal, and the values of the Clausius-Clapeyron relation along with the latent heat of the transformation are extracted.

Paper

Martensitic and reverse martensitic phase transformations are the physical processes that are responsible for the unique shape memory and superelasticity properties of shape memory alloys (SMA) [1]. These transformations do not involve a concentration change and can therefore be characterized by stress-temperature ($\sigma - T$) phase diagrams. Although phase diagram is a thermodynamic concept that is usually associated with a non-hysteretic phase transformation, it can be used in this context in cases where the macroscale resistance of the material to the phase transformation is constant [2]. In such cases, the $\sigma - T$ curves of the martensite to austenite ($M \rightarrow A$) and austenite to martensite ($A \rightarrow M$) transformations are separated by a constant distance in the $\sigma - T$ space and their slopes follow the Clausius-Clapeyron Equation [2]:

$$\frac{d\sigma}{dT} = \frac{L}{T_0 \Delta\varepsilon} \tag{1}$$

Here, L is the specific latent heat, T_0 is a reference temperature of the phase transformation and $\Delta\varepsilon$ is the strain change due to the phase transformation.

Stress-temperature phase diagrams of shape memory alloys constitute a design tool for SMA based actuators [3–5]. In addition, they are used as a basic input for macroscopic phenomenological models under quasistatic conditions [4–6]. In particular, finite element models of SMA's thermo-mechanical response rely directly on this information [4,5,7]. Models for the thermo-mechanical response of SMA at higher rates of operation also use the $\sigma - T$ diagram as a baseline [8,9].

At the microscale, the mechanism by which the phase transformations proceed are closely related to the microstructure that is formed near the phase boundaries (PB) [10–13]. The complicated evolution of the microstructure during the propagation of the PB results in a jerky motion that is composed of numerous local and temporary discrete impulsive events [14,15]. These microscale events determine the macroscale resistance of the material to the phase transformation. Therefore, it is essential to study the evolution of the microstructure simultaneously during a measurement of the $\sigma - T$ curves.

To this day, the $\sigma - T$ phase diagrams were obtained by measuring stress-strain ($\sigma - \varepsilon$) curves at different constant temperatures. Each of those $\sigma - \varepsilon$ curves provides only two data points in the stress-temperature space, $(\sigma^{A\rightarrow M}, T_{exp})$ and $(\sigma^{M\rightarrow A}, T_{exp})$. Here, T_{exp} is the temperature at which the corresponding $\sigma - \varepsilon$ experiment was carried out and $\sigma^{A\rightarrow M}$, $\sigma^{M\rightarrow A}$ are the plateau stresses for the forward and reverse phase transformations, respectively [2,16]. This procedure provides several discrete data points, rather than continuous curves. In addition, because the measured $\sigma - \varepsilon$ curves do not always display an ideal plateau, the choice of $\sigma^{A\rightarrow M}$ and $\sigma^{M\rightarrow A}$ is somewhat subjective. In summary, the procedure for obtaining phase diagram from individual mechanical loading tests requires the performance of several different experiments and provides a phase diagram that is based only on few discrete data points, rather than a continuous curve.

In this paper we present a new method which allows the direct measurement of the full $\sigma - T$ phase diagram in a single experiment. Moreover, the developed experimental setup enables simultaneous visualization of the microstructure evolution throughout the stress-temperature measurements via an optical microscope. The proposed method provides continuous stress-temperature-microstructure data

* Corresponding author.
E-mail address: eilonfa@technion.ac.il (E. Faran).

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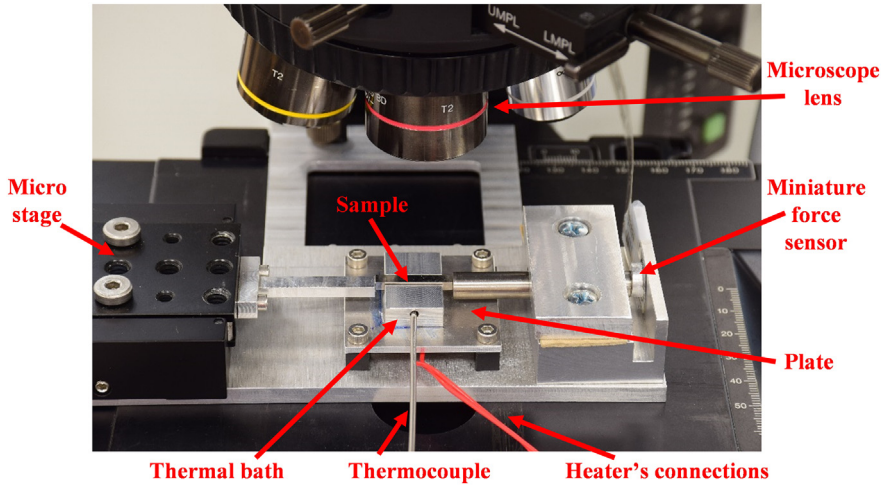

Fig. 1. The experimental setup used for direct measurement of the $\sigma - T$ phase diagram of a SMA sample.

that enable detecting local and temporary events, which are typical to the jerky nature of the phase transformation [14,15].

The experimental setup is presented in Fig. 1. The tested sample is placed at the center of an aluminum thermal bath with a U-shaped cross-section that surrounds the sample from three sides but enables the observation of the sample's top surface. The thermal bath rests on a 2mm thick aluminum plate. A thin Kapton® insulated flexible heater (OMEGA®) is glued to the plate's bottom side. The heater foil can reach temperatures of up to ~200°C. The temperature of the thermal bath is measured by a thermocouple, which is inserted into a designated bore in the thermal bath (see Fig. 1). A preliminary calibration experiment indicated that the difference between the temperature at the top surface of the sample and that measured inside the thermal bath is smaller than 2°C, at a measured range of 25°C − 85°C.

The tested sample is constrained in compression between two aluminum rods. The left rod is connected to a linear precision stage equipped with a micrometer and allows the adjustment of the initial compression along the longitudinal direction of the sample. The right rod is free to slide and pushes against a miniature force sensor (FlexiForce, type A-201) with a thickness of 0.2mm, which is located between the rod and a rigid aluminum wall. The entire experimental setup is placed under an optical microscope.

We demonstrate the capabilities of the developed method by measuring the $\sigma - T$ phase diagram of $Ni_{50}Mn_{28.5}Ga_{21.5}$ 10 M single crystal, produced by AdaptaMat LTD, with dimensions of 20mm × 3mm × 2.5mm. At room temperature this material is completely at the martensite phase. Prior to the experiment, the crystal was compressed along its 20mm long axis. This procedure brought the sample to a state of a single variant, in which the short crystallographic axis of the nearly tetragonal unit cell (c-axis) is parallel to the compression axis.

The experimental procedure for measuring the phase diagram is divided into two parts: (a) nucleation of austenite-martensite PB due to initial heating of the sample and (b) progression of the phase transformation by the motion of existing PB under constant heating and cooling rates. The $\sigma - T$ curves are measured during part (b).

Part (a) starts with controlled compression of the sample, at room temperature, by adjusting the micro-stage. This preload is necessary to prevent an abrupt phase transformation of the entire sample upon heating. Thereafter, the sample is slowly heated until a PB is formed. Nucleation typically occurs under non-equilibrium conditions and therefore the temperature required for nucleation is higher than the temperature required for the motion of an existing PB. Therefore, after the nucleation, the stress and temperature are gradually decreased by releasing the micro-stage and reducing the power supplied to the heater, such that the initial stress and temperature values, at which part (b) starts, are as small as possible. For the Ni-Mn-Ga sample studied in this work, at the end of part (a) there were typically two adjacent PB located near the sample's center and separated 2mm − 4mm apart from each other (see Fig. 2).

During part (b) of the experiment the micro-stage position is kept fixed, such that the overall length of the sample is constant. This constant strain condition results in the following relation between the austenite volume change Δv^A and the axial stress σ:

$$\Delta v^A \cdot \Delta\varepsilon + \sigma/E = 0 \tag{2}$$

The first term in Eq. (2) represents the strain change due to the phase transformation, where $\Delta\varepsilon$ is positive in our experiment. The second term represents the elastic strain, where E is an effective Young's modulus and σ is the developed compression stress (i.e., negative) due to the phase transformation. We note that the effective Young's modulus E is influenced by the compliance of the experimental setup, as well as by possible dependence of the sample's Young's modulus on Δv^A and the temperature [17]. Eq. (2) can be used to analyze local and temporary events. For example, a local barrier for the transformation may result in a temporary pause of Δv^A and in accordance a nearly constant value of σ (due to small possible changes in E) while T continues to increase. A subsequent overcome of the barrier may result in a rapid change of Δv^A until σ returns to the equilibrium value that is related to T. Such a behavior is expected to form steps on the $\sigma - T$ curve.

Part (b) of the experiment begins with heating of the sample to induce $M \rightarrow A$ phase transformation that is accompanied by an increase of the compression stress, in accordance with Eq. (2). The transformation proceeds by the propagation of the existing PB towards the edges

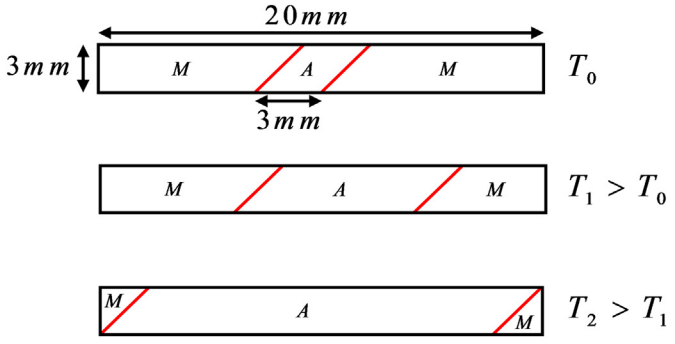

Fig. 2. Illustration of the locations of the austenite-martensite phase boundaries (red) during different stages of the experiment. A and M represent the austenite and martensite phases, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)