Study of Shape Memory Alloys and the Phase Transition by DMTA and DSC Measurements

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Abstract
The aim of this research is the examination of the DMTA technique to measure the phase transition temperatures of shape memory alloys. These parameters are difficult to detect by traditional material testing techniques, meanwhile quick and reliable results can be provided by DMTA measurements.

Keywords: DMTA, phase transition, shape memory alloy, complex modulus, Nitinol.

1. Introduction
The shape memory alloys are shape memory intelligent materials. Their main functional property is the shape memory effect. It means, that a permanent macroscopic deformation of the specimen can be ceased after heating it above a certain temperature. The basis of the shape memory effect is the martensitic phase transition. The alloys are usually adjusted such, that they have martensitic phase in room temperature and their austenitic phase transition temperature is below 100 °C. When the deformed martensitic specimen is heated to its austenitic phase transition temperature, the grid transformation occurs, and the specimen recovers its original shape.

The problem of the production of shape memory alloys is the determination of the phase transition temperatures. The most important parameters of manufacturing these materials are the temperature range of shape memory effect and superelasticity.

There have been many examples of measuring transition temperatures of metallic materials by DSC method, which was technically designed for plastic specimens. This method has also become well known in metal technology for the last ten years. However, the DSC measurement can only provide relevant results in case of higher heating speed, which worsens the reliability of the results.

The other possible method to measure the phase transition temperatures of such materials is DMTA. Our goal was to study if more exact results can be recovered by DMTA measurements. The other part of our research was studying a special kind of shape memory alloy, the Nitinol. It can be used as a material of a new kind of actuator in prosthesis hands. These kinds of actuators are compact and silent, so they make it possible to build a moving prosthesis. To demonstrate what these actuators are capable of, a prosthesis hand was built based on our design. [1, 3, 4, 7, 8]
2. Method

When a sinusoidal load is applied to a viscoelastic material (e.g.: polymers), a loss angle will exist between the load and the responding deformation. Its value will be between 0° and 90°. The complex modulus (E*) expresses the properties of viscoelastic materials. The real part is the storage (E’) and the imaginary part is the loss modulus (E’’). The ratio of loss and storage modulus (E’’/E’) is tanδ which describes the elasticity and the plasticity of the samples. When the value of tanδ is high the material acts more viscous, while low tanδ means a more elastic behaviour. The DMTA monitors the changes of tanδ as a function of temperature. The main unit of a DMTA equipment is the furnace, the moving and the fix clamp. During the measurement the temperature changes in the furnace, while stress activation is applied on the sample with the moving clamp. That creates a direct link between the material’s chemical structure and mechanical properties. Meanwhile the drawback of DSC is that the change of mechanical properties can not be monitored. [2, 6, 7]

3. Measurements results

The specimens were CuAlNi and CuAlNiMnFe rods in martensitic phase. The rods outer diameter were 2 mm and they were cut into 20 mm long pieces. A TA instruments Q800 DMTA and a TA Instruments 2920 Modulated DSC equipments were used in our experiments. Our research focused on the DMTA measurements and the DSC results came from the University of Miskolc, Faculty of Material Science. The test frequency was 5 Hz, the amplitude was 30 µm and the heating rate was 3 °C. The applied temperature range was between 0 and 200 °C. [2,] Figure 2 shows the DMTA measurement results for CuAlNi specimen. There is a local tanδ peak in the heating cycles at about 80 °C, which we consider to be in the austenitic transition temperature range. Other peaks can be seen in the cooling cycles at about 100 °C, which is considered to indicate the martensitic phase transition. The change in the tendency of tanδ indicates that these shape memory alloys showed "viscoelastic" behaviour.

![Figure 1. The first prototype of the prosthesis hand which is actuated by Nitinol springs](image)

![Figure 2. Recurrent heating and cooling cycles on CuAlNi specimen indicating the phase transition temperatures of the alloy](image)
Figure 3 shows the DMTA measurement results for CuAlNiMnFe specimen. The aim of this measurement was to compare the results to DSC measurement results which were made in the University of Miskolc, Faculty of Material Science. The tan delta peak is at about 78 °C in heating cycles and 95 °C in cooling cycles. The DSC curve of the same alloy showed the phase transformation approximately at the same temperatures. These results make DMTA measurement a potential new technique in the investigation of metals. Discrepancies between the peaks of cooling cycles in case of DMTA and DSC measurements have been found. The reason is, that the specimen used in DMTA is approximately 50 times heavier than those used in DSC, which affects the thermal conductivity. We used liquid nitrogen to cool down the specimen in the DMTA furnace, which might have had an influence on the results, too. [5]

Figure 3. DMTA curves of CuAlNiMnFe specimen showing local peak at 95 °C during heating and at 78 °C during cooling

Figure 4. DSC curves of CuAlNiMnFe specimen showing local peak between 87 °C and 105 °C during heating and between 82 °C and 92 °C during cooling [2]

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