Ultrasonic attenuation by dislocation formation in NiTi shape memory alloys

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Abstract
Dislocations are induced in polycrystalline NiTi shape memory alloy by thermal and mechanical cycling. To study the dislocation formation the ultrasonic velocity and the attenuation was measured at room temperature as a function of microwave frequency in the range 10 to 60 MHz. The experimental data are analysed in the frame of the vibrating-string-model. The fits yield length and density of the dislocations in the thermally treated and in the non-treated samples. Depositing the sample at 300 K for a couple of days leads to a reduction of the dislocation density accompanied with a shift of the transition temperature.

Introduction
The alloy NiTi at near-equiatomic composition presents one of the most promising shape-memory systems for application in medical areas as well as in technical disciplines. The martensitic phase transformation of NiTi which provides the base of the shape memory effect has been investigated very thoroughly in single crystals by several groups using x-rays, neutron scattering and ultrasonic techniques [1]. The phase transformations and by this the functionality of devices based on the shape memory alloy NiTi are strongly affected by presence of lattice defects. Beside impurities such as carbon, dislocations and precipitates in Ni-rich alloys are most important. Particularly, in the course of thermal and mechanical cycling processes dislocations are formed which impede the transformation. Propagation and attenuation of ultrasonic waves provide a sensitive tool to explore the creation and destruction of the dislocations. Here we report on ultrasonic experiments performed on polycrystalline NiTi alloy samples which had been subjected to annealing and thermal treatments [2].

Experimental
Ultrasonic velocity and attenuation of the longitudinal waves have been measured by pulse echo technique in the frequency range from 2 MHz to 60 MHz. Cube like shaped samples cut from rods were prepared for the investigation of the bulk elastic properties. To eliminate precipitates the samples had been first heated in a furnace in an inert gas atmosphere up to 1000 K and then quenched in water. Some of the solution quenched samples had then subjected to thermal cycling in the range –60 °C to + 40°C. The frequency dependence of the ultrasonic velocity and the attenuation was measured after quenching and after each thermal cycle and after the last thermal cycle the ultrasonic response was monitored as a function of time during several days. The ultrasonic experiments were complemented by DSC measurement to determine thermodynamic parameters such as transition temperatures.

Results and Discussion
The DSC measurements show that with increasing number of thermal cycles the phase transition temperatures of NiTi are continuously shifted to lower temperatures. However, when the sample is retained at room temperature a couple of days between two thermal cycles the phase transition temperature again increases. This behavior is in agreement with the creation and annealing of dislocations as observed by the frequency dependent ultrasonic measurements. Fig. 1 shows the ultrasonic attenuation of the longitudinal elastic waves at room temperature as a function of the ultrasonic frequency. Results are displayed for the solution quenched sample and for the sample which had been subjected to three thermal cycles. The lines represent best fits of the measured data using the vibrating-string-model. In the scope of this model dislocation pinned at their two ends are considered as a string with a resonance frequency and a damping constant [3]. The sound waves which interact with the vibrating string are attenuated and their velocity is modified. Both, the change of velocity and the attenuation are a function of the length and of the

density of the dislocation. The adjustment of the vibrating string model to the measured data of the attenuation and the variation of the sound velocity (not shown here) yield lengths of dislocations of the order of 15 µm and densities varying from 1.3 10^4 /mm^2 to 2,2 10^4 /mm^2 for the quenched sample and the thermally cycled sample, respectively

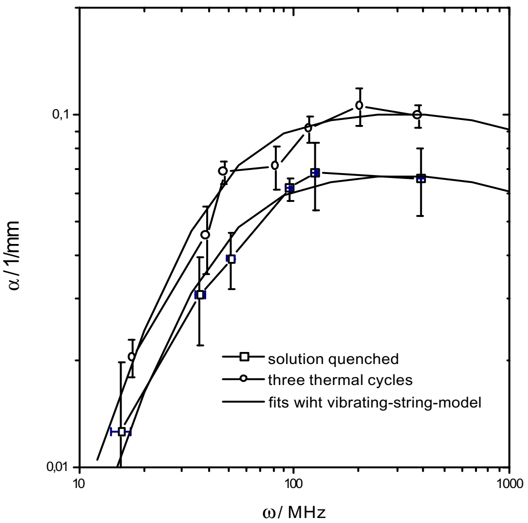

Fig. 1: Frequency dependence of the attenuation of the longitudinal modes measured a room temperature. Experimental data from a polycrystalline NiTi sample after solution quenching (squares) and after three thermal cycles (circles). Full lines are fits according the vibrating-string-model.

When the thermally cycled sample is deposited at room temperature the attenuation coefficient α gets reduced with time. Over a period of several days the attenuation measured at a frequency of 10 MHz decreases about 30 % reaching an asymptotic behaviour above 2 days. This observation is explained by a thermally activated annihilation of dislocations in agreement with the measured exponential decay with time of the attenuation coefficient α. However, the attenuation coefficient α levels off at a higher value than that of measured in the solution quenched sample. The difference between the solution quenched sample and the thermally relaxed sample is accounted for the influence of point defects where dislocation can be pinned and therefore are not able to be annihilated by the recombination with another dislocation. The ultrasonic results support the interpretation of the DSC data where a shift of the phase transition temperature with storage time was observed. Obviously the presence of dislocation imposes a reduction of the phase transition temperature.

Beside thermal treatment also an uniaxial pressure has an observable influence on the dislocations. Applying the pressure the attenuation of the longitudinal waves first increases in order to remain constant above a pressure of 100 MPa. The increase of the damping in the low pressure regime is most probably due to the depinning of dislocations. The depinning leads to more extended dislocations which are more effective in attenuating the sound wave. Simultaneously with the increase of the attenuation also the sound wave velocity increases in accordance with the prediction of the vibrating-string-model.

Conclusion

Frequency dependent ultrasonic experiments reveal an increase of the dislocation density by successive thermal cycling. The change of the ultrasonic attenuation and sound velocity levels off after three thermal cycles. Time dependent relaxation effects are observed for the thermally cycled sample stored at room temperature. With time the attenuation decreases levelling off at a slightly higher α-value than in the quenched sample. Simultaneously, the phase transition temperatures recover. These observation are attributed to reduction of the density of dislocation by recombination at ambient temperatures. Evaluation of the frequency dependent ultrasonic data on the basis of the vibrating-string-model provides a means to determine quantitatively the length and the density of the dislocations in NiTi shape memory alloy.

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References

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