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Calorimetric study of avalanche criticality in the martensitic phase transition of $Cu_{67.64}Zn_{16.71}Al_{15.65}$

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Abstract The first-order diffusionless structural phase transition in $Cu_{67.64}Zn_{16.71}Al_{15.65}$ is characterized by jerky propagation of phase front related to the appearance of avalanches. In this work we describe a full analysis of this avalanche behaviour using calorimetric heat-flux measurements and the results are compared with acoustic emission (AE) measurements.

Introduction

As is well known first order phase transition in martensitic $Cu_{67.64}Zn_{16.71}Al_{15.65}$ is a special kind of phase transition because it is widely in temperature with a large coexistence interval of around forty degrees. Recently it has been shown [1,2] that the phase transition progress with two regimens: a smooth front propagation that represent the largest part of the transformation enthalpy and the crackling noise related to the progression of domain boundaries [3] that is related to the appearance of avalanches, where their statistical analysis leads to a power law. In this work the influence of the scanning temperature rate in the forward (on cooling) and reverse (on heating) transitions is analysed and the exponent of the power law is obtained and it is compared with the one obtained by AE measurements.

Experimental results

The sample used in the calorimetric measurements had a flat surface of 0.94 cm^2, a thickness of 0.389 cm and a mass of 2.6503g. The sample for AE measurements was much smaller with a dimensions of approximately $0.5 \times 0.5 \times 0.1$ cm^3 and a mass of 0.185g, both belong to the same matrix, that was prepared in "Universidad del Centro de la Provincia de Buenos Aires, Tandil, Argentina".

Measurements of heat flux (Differential Thermal Analysis, DTA trace) were performed using a high-resolution conduction calorimeter that has been described before [4]. The most important characteristic concerned to this work is that the heat flux resolution is better than 0.1 μW and the scanning temperature rate, $r = dT/dt$, may be as low as 0.001K/h. The measurements of heat flux have been done at 0.26, 0.27, 0.04 and 0.01 K/h on cooling and at 0.29, 0.04 and 0.005 K/h on heating. In fig. 1 the heat flux for the highest temperature scanning rate is represented in a wide temperature interval.

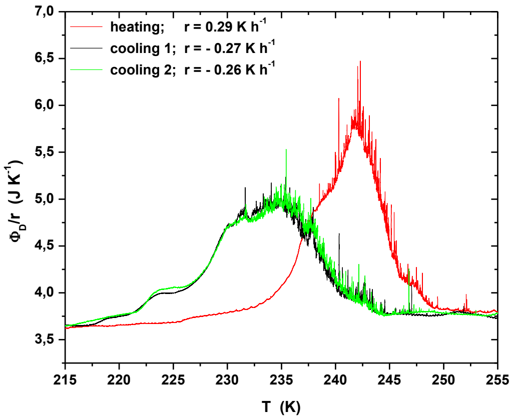

Figure 1. Heat flux over the scanning temperature rate obtained on cooling and on heating at approximately 0.27K/h

The latent heat has two contributions. The first is related to the smooth phase front propagation and the second arises from the spikes superimposed to the smooth background.

In figure 2a the heat flux on cooling for r = 0.27 and 0.04 K/h is represented. Both curves are not equivalent, but when the heat flux is normalized, that is, it is divided by the rate, r, the curves are very similar, figure 2b. This means that the smooth background is reproducible for different scanning temperatures rates. The same result is obtained when the experiment is performed on heating.

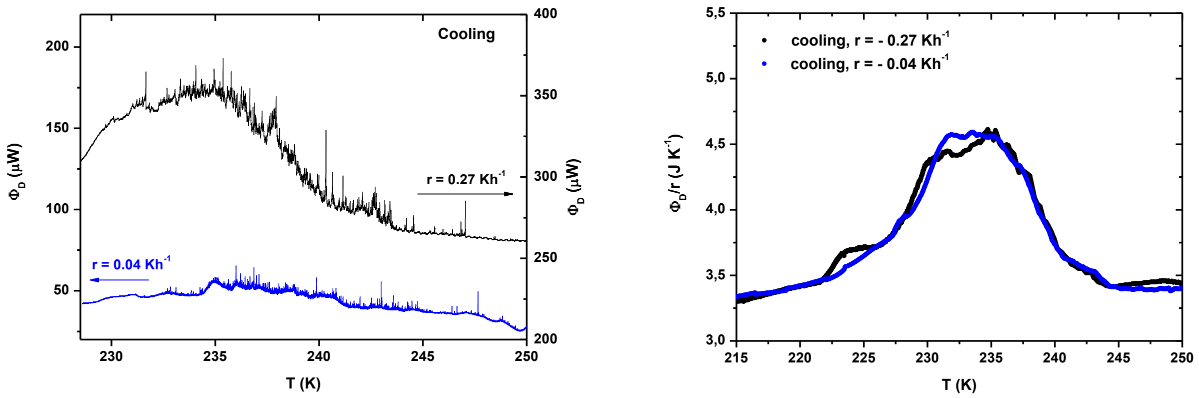

Figure 2a (left). Heat flux obtained on cooling at 0.27 and 0.04 K/h. Figure 2b (right). Normalized heat flux, without peaks, over the scanning temperature rate, for the same scanning temperature rate as in Fig. 2a.

The integral of ϕ/r, after appropriate baseline subtraction, leads to the excess enthalpy [1]. The total transition enthalpy is 370 ± 10 J/mol and is, within experimental errors, identical for heating and cooling and independent of r. The jerk enthalpy is <3% of the total and restricted to a smaller temperature interval [2].

In order to analyse the statistic of the peaks, the height of each peak was determined with respect to the smooth variation in the background. In figure 3 the peaks obtained on cooling for scanning temperature rate of r = 0.26, 0.04 and 0.01K/h, and for r = 0.29, 0.04 and 0.005 K/h on heating are represented. These figures show that the peaks on heating are higher than on cooling. This fact is particularly important for very low scanning temperatures rates, which indicates that the kinetics is not the same for both process. This may be related with the fact that on cooling, the forward transition goes from a single-domain austenitic crystal to a multidomain martensitic structure, so that the sample has less domain boundaries that can act as intrinsic defects and nucleate avalanches.

In the contrary, in the reverse transition the martensitic fraction decreases by the shrinkage of domains going form a multidomain to a single-domain structure, so the intersections between domain boundaries are bigger.

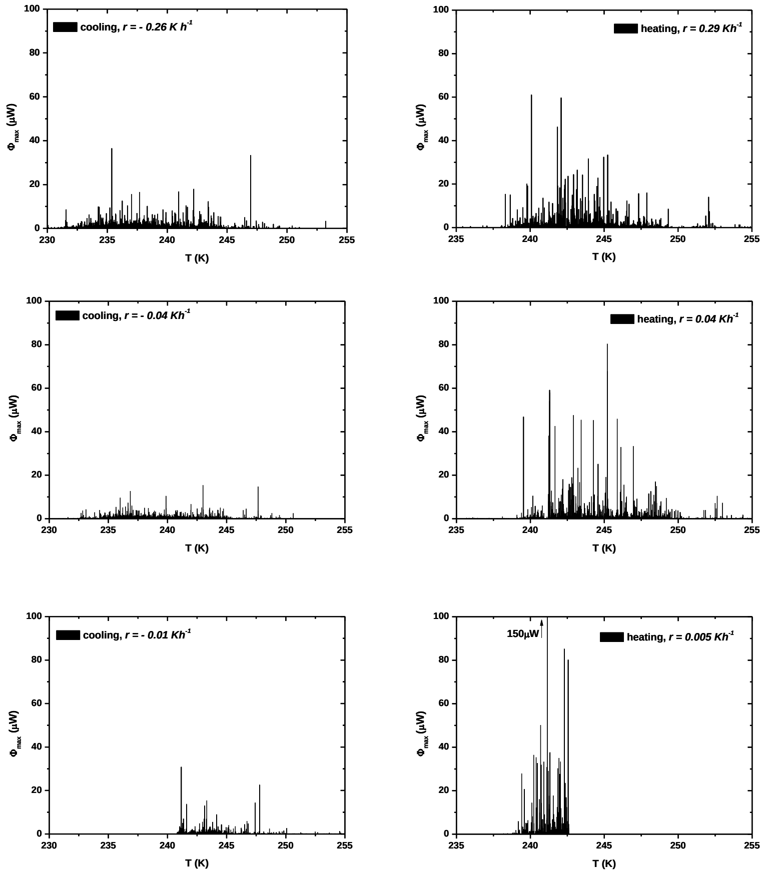

Figure 3. Peaks obtained on cooling and on heating for three different scanning temperature rates.

Taking into account that the response of the calorimeter device to a heat flux pulse in the sample is represented by a single-exponential decay whose time constant is larger than the characteristic time of the avalanches, the height of the peaks is proportional to the energy of each pulse:

$$E = \int_{\Delta t} \Phi(t)dt \approx \Phi_{max}\Delta t \propto \Phi_{max}$$

Acoustic Emission measurements, AE, was detected using a piezoelectric transducer, acoustically coupled to the sample. Hits were recorded during cooling and heating ramps between 270 and 215 K at rates in the range 1 - 0.5 K/min. Details of experimental method are described in reference [5].

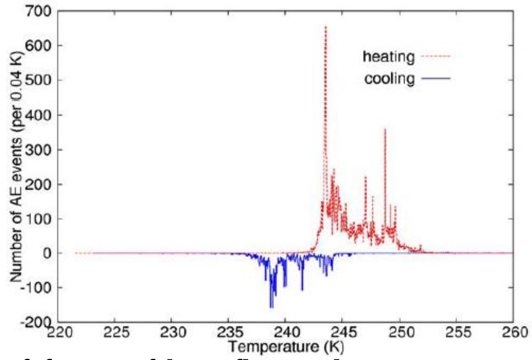

Figure 4. Acoustic emission activity as a function of temperature obtained on cooling and on heating

Inspection of figure of heat flux and AE measurements leads to the conclusion that both features have the same physical origin.

Avalanche Statistics, Maximum Likelihood (ML) Method

The jerks are analyzed statistically, with the peaks heights proportional to the energy per jerk E. In order to obtain the estimation of the power law exponent, we have performed Maximum Likelihood (ML) fits of a power-law probability density:

$$p(E)dE = \frac{E^{-\varepsilon} dE}{\int_{E_{min}}^{\infty} E^{-\varepsilon} dE}$$

where P(E)dE is the probability to observe an avalanche with energy E in an interval between E and E+dE and where the integration covers the full energy range. The lowest accessible energy per jerk is 1.5×10^{-5} J.

The expected dependence of the exponent, ε, with the cut-off E_{min} has been studied in detail in reference [6]. One expects that the exponent increases when the cutoff is increased while a plateau is seen at the best estimation of the exponent. Figure 5a shows the behaviour of the fitted exponent for calorimetric data on heating, and Figure 5b for the calorimetric data on cooling.

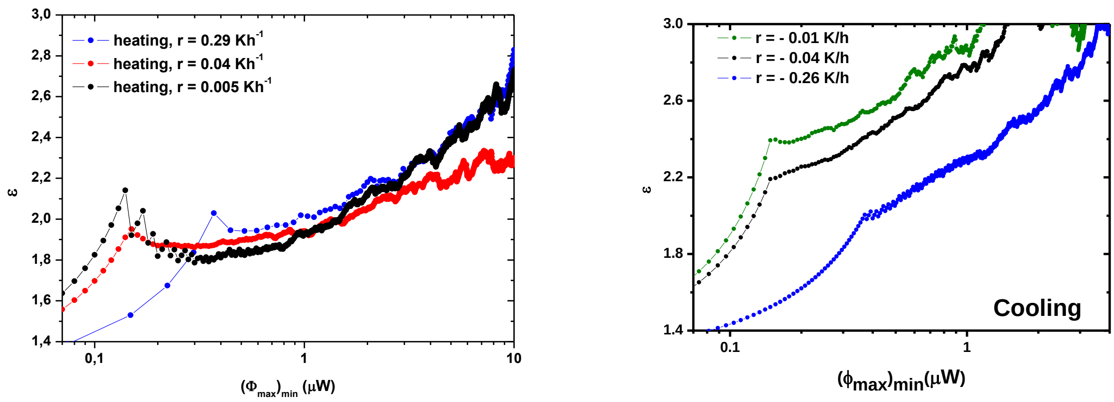

Figure 5a (left). Statistical analysis in the Maximum Likelihood method obtained on heating and, Figure 5b (right), on cooling.

On heating, figure 5a, a plateau is obtained with an exponent of ~1.8, being the plateau enhanced when the temperature scanning rate is lower. ML method for AE measurement gives similar results for the fastest curve on heating obtained by calorimetric measurement, as is shown in Figure 6.

On cooling, figure 5b, no plateau is obtained, neither for ultra low scanning temperature rate, indicating that the energies for the forward transition do not follow a power law.

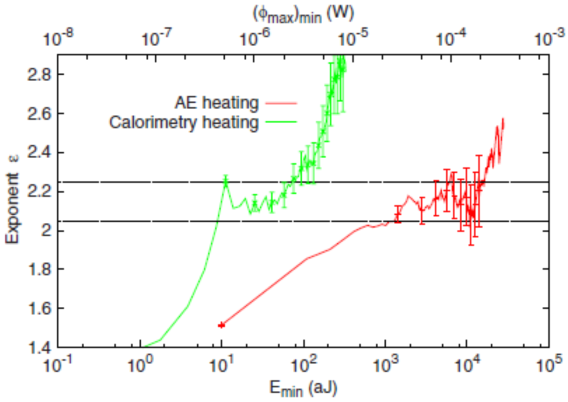

Figure 6. Power law exponent as a function of integration interval for calorimetric data on heating and for AE measurements.

Summary

Three different scanning temperature rates have confirmed that the phase front of the transition in $Cu_{67.64}Zn_{16.71}Al_{15.65}$ is developed in two regimens, a continuous background and the avalanche jerks. The smooth background is reproducible even at ultra low scanning temperature rate. The jerks are well defined when the scanning temperature rate is lower, so that the statistic analysis is more reliable and the plateau obtained on heating by ML method is enhanced. Nevertheless on cooling no plateau is obtained even for the smallest temperature scanning rate.

Although that avalanches seen in calorimetry are vastly more energetic than those seen in AE, the statistical analysis is identical for both experimental techniques, it becomes clear that the full range of the power law extends over several decades.

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