OPTICAL MICROSCOPY APPLIED TO THE DETERMINATION OF Cu–Al–Be SHAPE MEMORY ALLOYS PHASE TRANSFORMATION TEMPERATURES

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ABSTRACT
Polycrystalline specimens of Cu-Al-Be shape memory alloys were homogenized at 1123K during 12h and water-quenched at room temperature. The phase transformation temperatures were determined via optical microscopy technique, using a cooling/heating device attached to the microscope stage. Comparisons have been made with data obtained via differential scanning calorimetry. The data show that the former technique can be successfully applied to the determination of typical transition temperatures occurring in shape memory alloys. As far as sample related problems are concerned, the former technique can also represent an advantageous alternative over the latter given the fact that the analyses can be performed in more representative area. The M_f and A_s temperatures precise detection depend on the chosen resolution of the optical microscope.

Key words: Martensitic transformations, austenite transformations, shape memory effect.

MICROSCOPIA ÓTICA APLICADA À DETERMINAÇÃO DAS TEMPERATURAS DE TRANSFORMAÇÃO DE FASES DE LIGAS Cu-Al-Be

RESUMO
Amostras de ligas policristalinas Cu-Al-Be com memória de forma foram homogeneizadas em 1123K durante 12h e temperadas em água à temperatura ambiente. . Com auxílio de um dispositivo para aquecimento e resfriamento da amostra, adaptado a um microscópio ótico, foram determinadas as temperaturas de transformação de fase e registradas as imagens a elas associadas. Os dados obtidos foram comparados com aqueles obtidos por calorimetria diferencial de varredura. Os resultados indicam que ó método ótico desenvolvido pode ser empregado com sucesso à determinação das temperaturas de transformação de fase em ligas com memória de forma. Levando-se em consideração problemas com as amostras a serem analisadas, o método ótico representa uma alternativa vantajosa ao método calorimétrico. Por exemplo, no método ótico é possível escolher uma área mais apropriada para análise, o que não é possível no calorímetro diferencial de varredura. A determinação precisa das temperaturas A_S e M_F depende da escolha da resolução do microscópio ótico.

Palavras chaves: Transformações martensíticas, transformações austeníticas, efeito memória de forma.

INTRODUCTION

Shape memory alloys are potentiality attractive in several applications, especially as sensors and actuators. The main characteristic of these materials resides in the shape recovery and in the superelasticity properties. Both phenomena are intrinsically linked to the reversible martensite-austenite phase transformations [1]. These transformations occur in a certain temperature range and the precise determination of the transformation temperatures has fundamental importance for their applications. The more commercially used alloys are Ni-Ti, Cu-Al-Ni and Cu-Al-Be. Among them, only Ni-Ti and Cu-Al-Be are capable to be used at low temperatures (T<273 K).

Polycrystalline Cu-Al-Ni alloys also show good shape memory alloy at temperatures below 273K. Nonetheless these alloys become brittle at low temperature [2-5]. Although Ni-Ti alloys stand out by displaying superior thermomechanical and corrosion resistance properties, the high cost of raw materials and processing constitutes the main factor that limits their applications. Cu-Al-Be shape memory alloys can be

used as alternative to Ni-Ti alloys given their relative low cost and superior performance at low temperatures (T < 273K). The main reason for Be containing alloys to be used at low temperatures resides in the fact that the introduction of only 0,1wt% of Be promotes a drop of 100K in the transformation temperatures [6]. One of the difficulties to determine the phase transformation temperature by differential scanning calorimetry technique is associated with eventual modifications in the transformations temperature caused by introduction of strain during the cutting of specimen and loss of Be caused by oxidation during the thermal treatments [6]. Another inconvenience in the methods usually used to determine the phase transformation temperature (calorimetry, resistivity changes) can be attributed to an imprecise determination of the start and finish temperature of the transformation, making it difficult to establish the temperature in which the formation of the first martensitic needle occurs. This work deals with the evaluation the potential use of optical microscopy applied to the determination of shape memory alloys phase transformation temperatures.

MATERIALS AND METHODS

Cu–11.8Al-0.6Be(%wt) alloy was cast at room temperature by inductive melting and poured in rounded chill mold with a diameter of 20mm and 70mm height. Samples of 15mm in height were taken from the ingots and homogenized during 12 hours at 1123K followed by water-quenched at 298K. The specimens were mechanically polished with 0.1µm alumina. The metallographic analyses were carried out by optical microscopy using the Zeiss-Axiotech instruments at different temperatures. Phase's transformation doesn't instantly occur on the whole sample's surface; consequently, it is possible that transformation's temperatures values may not be exactly the same in different sample's areas. In order to minimize this inconvenient, a small magnification of 80x (spot diameter around 5-6mm) was used.

For heating and cooling the specimen between 298K and 173K, at an estimated rate of 10K/min, a proprietary stage composed of two parts was constructed. The first part is the specimen holder located in the superior level of the device and the second one, in the inferior level, is the thermal camera for cooling and heating, Fig. 1.

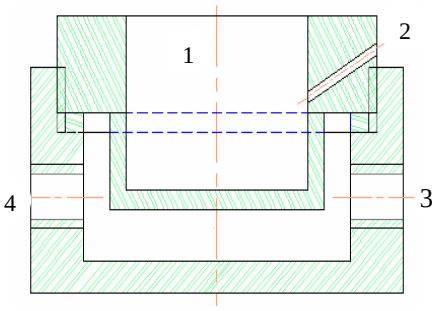

Fig. 1. Representation of the cooling/heating stage for metallographic analyses.

This stage is composed by the following parts:

1 - Specimen holder (immerged in ethyl alcohol).

2 - Thermocouple input.

3 - Inlet of the cooling and heating fluids.

4 - Output of the cooling and heating fluids.

The cooling and heating were obtained using both a liquid nitrogen and room temperature alcohol flow, respectively. The sample was immerged in ethyl alcohol to avoid the occurrences of the water condensates which causes the image to be blurred. The temperatures were measured at the surface of the samples, using a chromel-alumel thermocouple, connected to a milivoltimeter with exit to microprocessor.

The images were recorded using a digital camera attached to the optical microscope. A perfect synchronization between the shooting of the images and the temperatures reading assures the correlation between image and its corresponding transformation temperature.

The differential scanning calorimetry tests were performed in specimen with approximately 30mg, at a heating and cooling rate of 10 K/min, under nitrogen flow of 50ml/min using Shimadzu DSC-60 equipment.

RESULTS AND DISCUSSION

Fig. 2(a) to 2(d) and Fig. 3(a) to 3(d) shows the optical micrographs corresponding to the formation of the martensites during cooling and the martensite reversion transformation on heating, respectively.

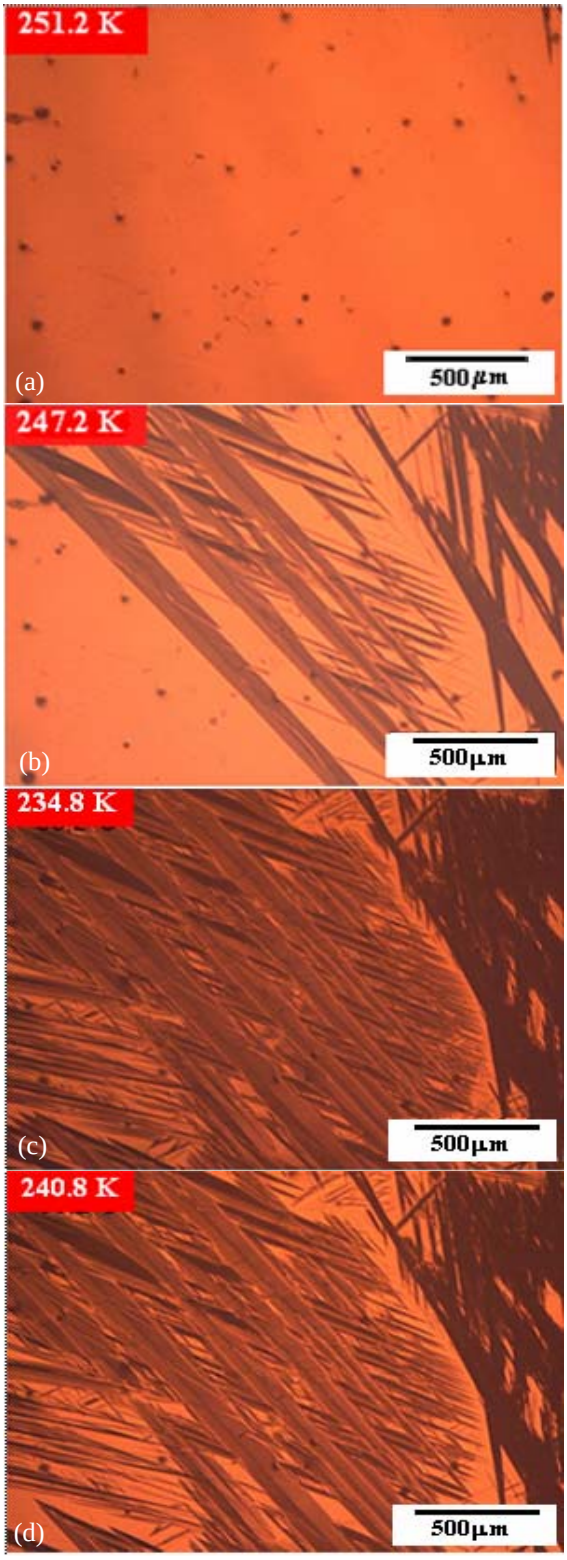

Fig. 2. Martensite formation sequence on cooling (a) martensite transformation start. (b) and (c) evolution of the martensite transformation. d) martensite transformation finish. The transformation temperature is marked in the superior left side.

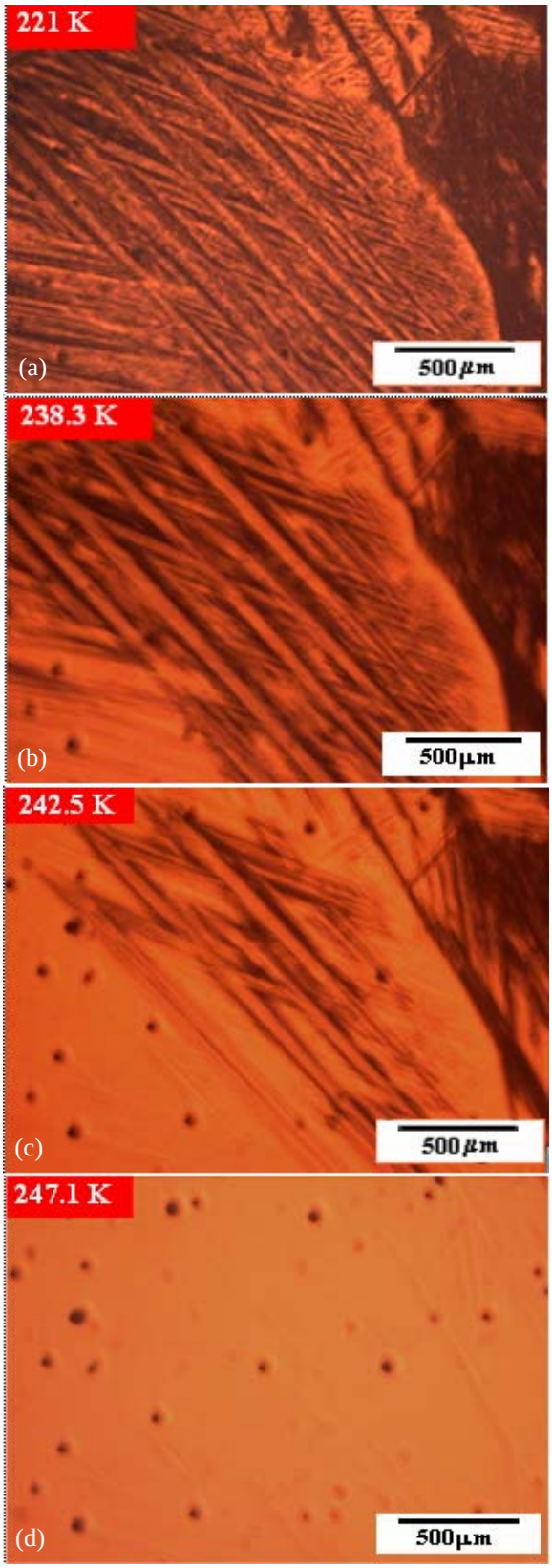

Fig. 3. Austenite formation sequence on heating (a) austenite transformation start. (b) and (c) evolution of the austenite transformation. d) austenite transformation finish. The transformation temperature is indicated in the superior left side.

In table 1, the start and finish temperatures for the martensitic and austenite transformation, obtained

through the metallographic method and differential scanning calorimetry (DSC), are listed. The DSC curve of the specimen is shown in the figure 4.

Table 1. Phase transformation temperature determined by the optical and calorimetry methods.

	Transformation temperature [K]			
Methods	M_S	M_F	A_F	A_S
Optical	251.2	234.8	247.1	221.0
DSC	242.2	217.2	253.6	231.6

The values determined by the two methods are quite close, except for the finish temperature of the martensitic transformation. The great difference in this value is caused by the fact that it is quite difficult to distinguish any visual morphologic alteration by the optical method when the martensite phase transformation is almost completed. This same reasoning is applied for the determination of the austenite transformation start, which occasionally presented quite close values in ours tests. The small variations found in other points (Mf and As) presented in the table I can be justified through the influence of certain parameters of the tests at the phase transformation temperature. For example, in the both methods the determination of the phase transformation temperature depends on the heating and cooling rate and the mass of the specimen, as well. In the optical method, these measures can vary with the positioning of the thermocouple in the specimen and with the heating and cooling rates. The specimen preparation can also induce small alterations of the results. For example, the presence of strain-induced martensite generated during mechanical polishing can induce martensite needle formation. Similarly, the cutting operation usually used for calorimetric method sample preparation can also alter transformation temperatures. Nonetheless, the specimen could also be electrochemically polished, reducing the detrimental effects caused by cutting. However, taken into account the usual small dimensions of the DSC samples, this procedure can hardly be achieved with success. Hence, optical microscopy might represent a better alternative as far as specimens size is concerned, given their suitability for electrochemical polishing. Also, the start of the martensitic transformation, which is easily identified by the optical methods, is hardly detected by DSC method due to the small quantity of release heat that take place during the formation of the first martensites needles or plates. Although the reflected area might be seen as small, one can increase the statistics of the analyses, observing other regions of the sample, which can not be done by DSC. In the optical case, anysotropies and processing defects can be precisely detected by scanning other sections of the surface, which is not the case in DSC analyses.

It is important to highlight that the image can becomes occur blurred for observations in very low temperatures (about 183 K). This inconvenience can be minimized covering the superior part of specimen holder with a polyethylene film. The simple heating of this film with a hot blower will avoid the formation of condensates.

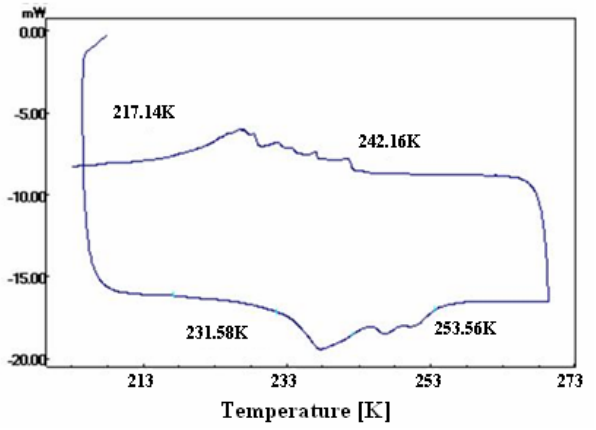

Fig. 4. DSC curve of the specimen with indication of the austenite and martensitic transformation start and finish temperatures.

CONCLUSIONS

The determination of the phase transformations temperatures of shape memory materials with low temperatures through optical observation can represent an alternative method. The variations of the phase transformations temperatures between the calorimetric and optical methods can be minimized through an appropriate control of certain parameters of the tests. More precise determinations of the phase

transformations through optical observation can be obtained with an electrolytic polishing of the specimen, using lower cooling rate and the substitution of the heating method to a resistive heating equipped with temperature control. The great advantage of the optical method resides in the facts that, through this technique, not only the determination of phase transformation start and finish temperatures is possible, but also the observation of the structure changes caused by the variation of the temperature.

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