A NEW METHOD TO DETERMINATE PHASE TRANSFORMATION IN SHAPE MEMORY ALLOYS: INFRARED THERMOGRAPHY

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ABSTRACT

In this article it is presented a shape memory alloy case, based on copper, namely Cu-Zn-Al, which is subjected to periodic mechanical traction. Traction is performed in conditions of normal temperature and pressure. The purpose of this article it is to study stress induced phase transformation. All tests are performed in same conditions. Transformation on which is based this effect occurs in two ways: by applying a stress or temperature variation. In this article it is studied stress induced phase transformation. The method to analyze the microstructure of an SMA is relatively new and it is based on tracking the evolution of temperature. After thermal analysis we can decide in which state is one alloy without any other supplier measures (DSC or electrical resistivity).
If our specimen will producing thermal energy when specimen is tensile he is austenitic. If absorbing heat during the first deformation is in martenitic state.

Key words: shape memory alloys, phase transformation, calorimetric measures, infrared camera

I. INTRODUCTION

Shape memory alloys (SMA) are a special group characterized by two main properties, shape memory effect memory defined as thermal and mechanical memory known as pseudoelasticity. Those alloys may have properties or mechanical behaviour and thermo-mechanical side of most important in terms of applicability is the double memory and effect of vibration damping (damping capacity).
Shape memory alloys based on Ni-Ti presents the best properties for most industrial applications. But their price is very high compared to shape memory alloys based on copper. In many applications of copper based alloys offer economical alternative to the Ni-Ti **[1].** Shape memory effect memory is defined as thermal and mechanical memory known as pseudoelasticity. In most materials, as in the

SMA, there is another type of thermomechanical coupling: thermoelastic coupling. Transformation on which is based this effect occurs in two ways: by applying a stress or temperature variation. The martensite austenite phase change produces latent heat contributing to an overall increase of sample temperature. After a phase change due to a stress action in a SMA at ambient temperature, thermomechanical coupling plays an important role in the sample mechanical response. This change in temperature influences the kinetics of transformation, even more; the last one can be induced by temperature.

The connection between temperature and phase change were studied in detail by many people interested in this phenomenon [2]. Determining of phase transformation temperatures in shape memory alloys with differential scanning calorimetric or DSC is a accuracy method. This is the most important method which is used also to development of successful SMA actuator [3]. With this we can prove that one shape memory alloy is find in martensitic or austenitic state and find exactly what are values of Austenite finish (Af), Austenite start (As), Martensite start (Ms) and Martensite finish (Mf).

The level of irreversibility can be estimated from theoretical approaches based on the so-called crystallographic compatibility [4], [5]. Experimental assessments of the transformation irreversibility have been also proposed in the literature with a special emphasis on the influence of the chemical composition: measurement of the temperature-induced transformation hysteresis [6] and measurement of the damping properties [7]. The present work proposes to assess the level of irreversibility of the stress-induced transformation by using infrared (IR) thermography.

The reversibility of the austenite-martensite transformation occurring in shape-memory alloys is a key-point of the mechanical performance of these materials. Infrared thermography is employed in this study to measure the mechanical (intrinsic) dissipation produced by a SMA specimen under cyclic loading at constant ambient temperature. Several Cu-Zn-Al specimens with different chemical compositions are tested. The experimental procedure followed is first discussed. Results obtained clearly show that the nature of the phase involved (martensite or austenite at the unstretched state) strongly influence variation of temperature produced by the specimen.

II. EXPERIMENTAL PROCEDURE

The studied alloy was prepared using an induction furnace with graphite crucible in air using high purity metals. Chemical composition was determined using Foundry Master Spectrometer. The chemical analysis was performed using the spectrometer at several points on the sample surface, and then executed mediation for a more accurate analysis. Microstructure in cast state and after annealing was emphasized by scanning electron microscopy (SEM). To this purpose we used a microscope of type II Vega LMH produced by TESCAN using a secondary electron detector.

All materials were first elaborated to obtain a parallelepipedic ingot for each composition. The upper and lower surfaces where then machined to obtain flat and regular surfaces, whose thickness were nearly 4 mm. The specimens were then heated up to 750°C and hot rolled to obtain the desired final thickness, which was equal to nearly 1 mm. Three passes were necessary to reduce this initial thickness from 4 to 1 mm. The resulting sheet was finally cut to obtain the specimens that were tested. All specimens were finally tempered by heating up to 750°C and cooling by air up to ambient temperature. Square aluminum tabs were bonded on the ends of the specimens to prevent any slippage within the grips of the testing machine. The dimensions of the gauge section slightly changed from one specimen to each other, depending on the dimensions of the sheet where they were cut.

Processing of the data has been used in the analysis of polycrystalline SMA. This consists in processing temperature maps derived from the process that is subjected the SMA sample, maps recorded by a computer that is connected to the entire system. Data processing consists in analyzing the sample surface temperatures and extracting heat sources starting from the temperature maps. Variation of surface

temperatures of a SMA sample is obtained after subjecting it to periodic and cyclic traction, and here it will be noticed the relationship between elastic strain amplitude and temperature amplitude.

A. *Mechanical loading*

All tests were performed on the same tensile testing machine (MTS ± 15kN) and temperature (T0 = 22 ± 2° C). The basic procedure is the same for all tests. **Figure 1** shows this procedure:

- **Stage I**: In this step we imposed deformation ε_{macro} , the objective is to partially transform the martensite in sample. The macroscopic deformation speed is constant for all tests: $2x10^{-3}$ s^{-1}. Macroscopic final deformation is noted ε_{macro} and not vary from a test to another;
- **Stage II**: this corresponds to a waiting period before the imposed displacement. This procedure allowed the sample to return to room temperature. Indeed, during the previous round, the sample temperature increased due to the latent heat associated with phase change. For all tests in this study, three minutes have been used in practice, so that the sample to return to thermal equilibrium.
- **Stage III**: a small periodic displacement was imposed to the mobile parts of traction machine around the position reached in the previous step. Loading frequency and strain amplitude, they are noted as follows: f_L and $\Delta\varepsilon_{macro}$. For this step we will change strain amplitude to see influence of ampltitude of temperature in function of variation of strain amplitude.
  In practice, we used the following values for $\Delta\varepsilon_{macro}$: 0.025 mm, 0.05 mm and 0.150 mm and the same value for f_L = 13 Hz. During this phase was short for all tests in order to limit damage to fatigue: 10 seconds in practice.

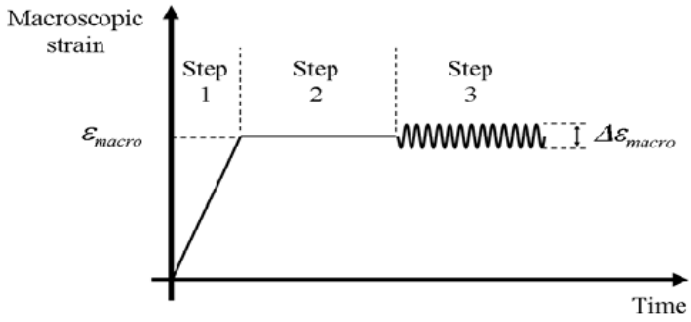

Figure 1 *Basic loading procedure*

B. Temperature measurement

To record sample temperature evolution during charge phase 3 an infrared camera Jade III-MWIR Cedip was used. This device has IR detectors characterized by 3.5 - 5 microns wavelength. Integration time used in these measurements is 1500 microseconds.
Thermal resolution is 0.02° C, and purchase frequency f_a = 150 Hz.

Purchase frequency should not be a multiple of the loading frequency for a better data treatment by discrete Fourier transformation.
Temperature map captured by infrared camera has a 80 × 60 pixels resolution. Spatial resolution is 0.36 mm (this corresponds to a pixel size). Surface sample "pursued" by the IR room was uniform painted with a matt black paint and opaque. This will cause a thermal emissivity close to 1. The pain was applied few moments before the test, in order to reduce the risk of its removal during the examination.

III. RESULTS AND DISCUTIONS

To detecting microstructure of study SMA it was used coupling between infrared camera and traction machine. This method informs us about evolution of temperature on the surface of specimen during the test.
Each specimen was subjected to three short displacement-controlled cyclic tests (frequency=13~Hz), the prescribed displacement increasing from one of the three stages to each other. For all specimens, this mean strain of the cyclic loading was equal to 1\%, 2\% and 3.2\% for each of the three stages, respectively.
Figure 2 show us one case of martensitic specimen where we enrigesterd a small negative variation of temperature (-0.3°C) at the surface of specimen because of thermoelatic coupling. This effect is characteristic only for martensitic specimens.

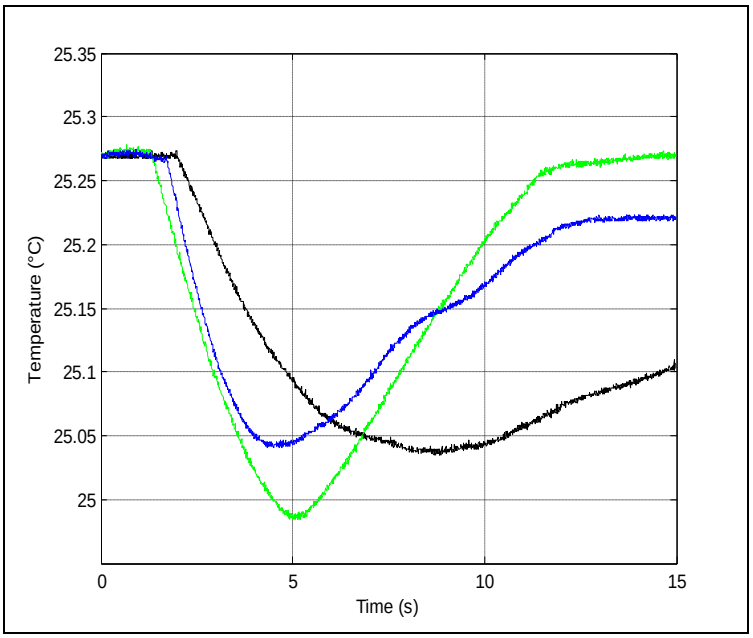

Figure 2 *Evolution of temperature during the initial charging for a martensitic specimen*

In **Figure 3**, it is represented martensitic microstructure of one Cu-Zn-Al shape memory alloys in quenched state of room temperature.

Alloy microstructure was studied at four different magnifications, respectively 200X, 500X, 1000X and 5500X, it highlights the auto accommodated oriented martensite variants obtained following treatment of hardening and martensite obtained directly from the casting material and quenched.

In the figure below are some which are highlighted microscopy of martensite variants of various shapes and sizes, grain boundaries and the intersection of three grains.

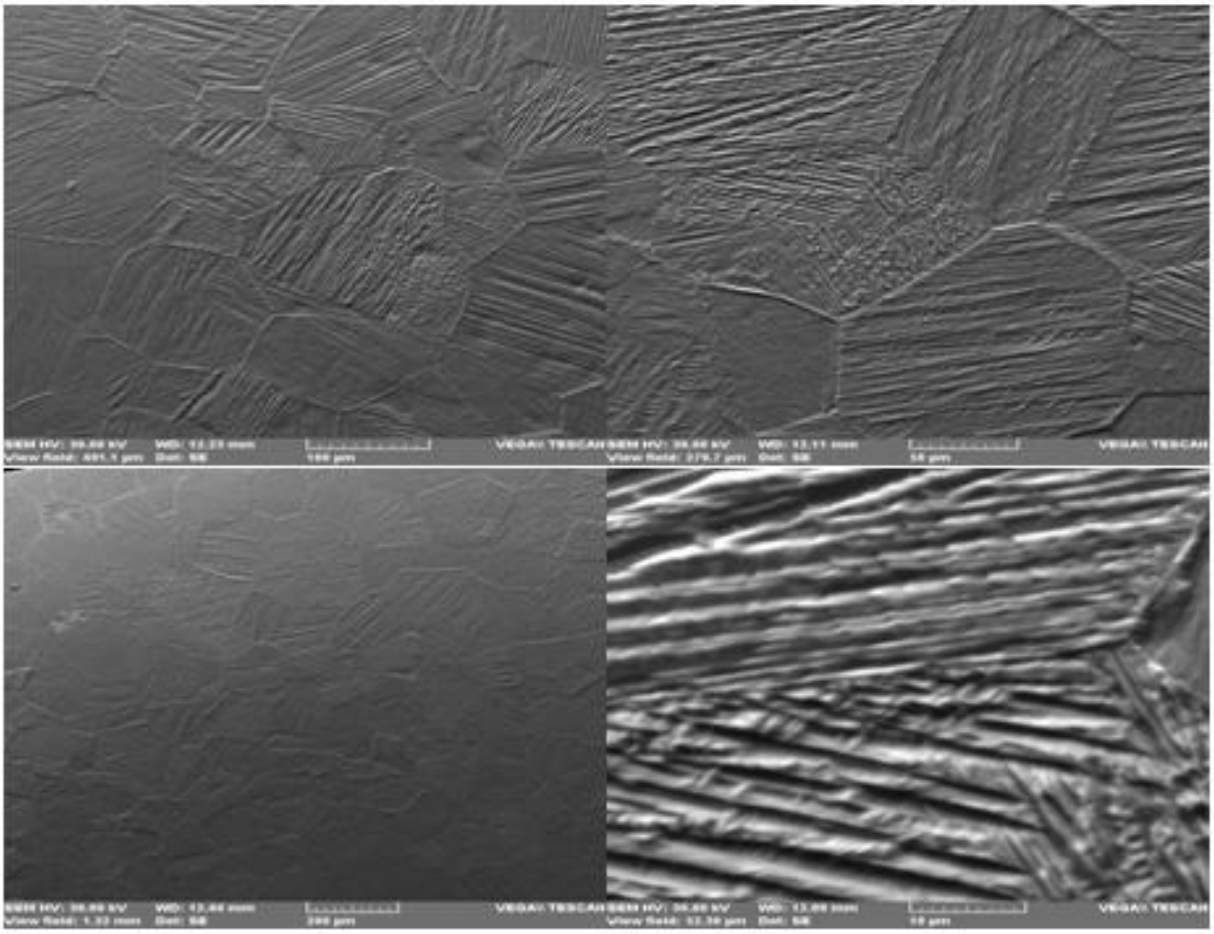

Figure 3 *SEM micrographs of memory shape alloy in quenched state at 200x, 500x, 1000x and 5500x*

Figure 4 show us one case of austenitic specimen. In this case thermal measures during the initial charging lead us to a higher positive variation of heat source (+3.5°C) at the surface of specimen. This is effect of latent heat producing during phase transformation austenite→martensite, where we obtained martensite induced by tension. This effect is characteristic only for austenitic specimens.

In terms to show that thermal result is correct we present micrographs of austenitic specimen. Alloy microstructure was studied at four different magnifications, respectively 200X, 500X, 1000X and 5500X.

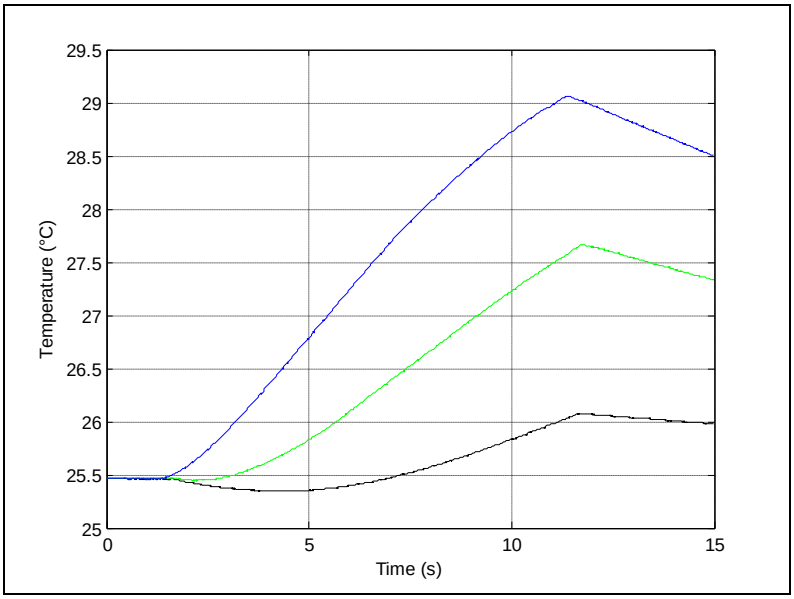

Figure 4 *Evolution of temperature during the initial charging for a martensitic specimen*

IV. CONLUSIONS

Study of shape memory alloys is of great interest today. Austenite and martensitic phase play a critical role in this study.
Transformation takes place during the release or absorption of heat.
This is highlighted in this article using relatively new analysis methods, namely infrared thermography.
The aim is to highlight the variation of temperature depending on the transformation austenite-martensite or marteniste reorientation.
By studding heat field on the surface of specimen we can determinate if one specimen is:

- Martensitic – low temperature variation→due to thermoelatic coupling;
- Austenitic- high temperature varaiation→due to latent heat producing by phase transformation.
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REFERENCES

[1] S. Stanciu: Shape memory materials (Materiale cu memoria formei) – Investigation methods and technical applications (Metode de investigație și aplicații în tehnică), 2009 ed. Universitas XXI, ISBN 978-606-538-004-2 (2009),p. 12-20

[2] Zeghdane S., Necib, K., Britah A.: Structural Characterization of Cu- 13.58%Al- 3.94%Ni (wt.

%) shape memory alloy elaborated by fusion, EPJ Web of Conferences 6,29001, 2010

[3] J.H. Mabe ; C.J. Yu ; E. Rosenzweig, A comparaison of the DSC measurement of shape memory alloys and the materials thermal characteristics in a large scale actuator, Smart Structures and Materials 2006: Active Materials: Behavior and Mechanics, 617011 (April 06, 2006)

[4] Cui J., and all. (2006), Combinatorial search of thermoelastic shape-memory alloys with extremely small hysteresis width, Nature Materials 5:286-290

[5] Chang S.H. (2011), Influence of chemical composition on the damping characteristics of Cu-Al-Ni shape memory alloys, Material Chemistry and Physic, 125:358-363

[6] Chrysochoos. Infrared thermography applied to the analysis of material behavior: a brief overview. QIRT Journal, 9(2):193–208, 2012. DOI: 10.1080/17686733.2012.746069

[7] D. Delpueyo, X. Balandraud, and M. Grediac. Applying infrared thermography to experimentally evidence martensitic microstructures in a Cu-Al-Be shape-memory-alloy single crystal. Materials Science and Engineering: A, 528(28):8249–8258, 2012