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A SHAPE MEMORY ALLOY CHARACTERIZATION THROUGH THERMAL ANALYSIS

BY

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Abstract: A shape memory alloy type Cu-Zn-Al was analyzed with different equipments for a thermal characterization. The alloy was investigated with a scanning electron microscope to determinate his microstructure and after that with equipments like a differential dilatometer (DIL), to establish the shape memory effect, a differential scanning calorimeter (DSC) for critical transformation points determination and a dynamic mechanical analyzer (DMA) for his mechanical properties.

Keywords: shape memory effect, calorimetric, dynamic mechanical analyzer.

1. Introduction

Thermal analysis represents the measurement of some physical parameter of a system as a function of temperature, usually measured as a dynamic function of temperature.

Thermal analysis represents an amount of investigation methods where physical and chemical properties of a substance, a substances mixture or some reaction products are measured as a temperature or time function. For technical realization of this determinations the analyzed product temperature is modify by a well fix controlled and establish schedule /1/. During temperature modification the physical property pursued is continuous measured the result being usually a graph automatically obtained (a thermograph) which contain the tracked property on vertical and temperature on horizontal. Chemical substances and materials generally present a series of characteristic points with temperature where take place specific transformations specific for each material like: melting or boiling point, solidification or optical exchanges.

The object of technical thermal analysis represents the measurement of these transformations with temperature using different equipments. The primary types of thermal analysis are TG (Thermo-gravimetric) analysis: weight, DTA (Differential Thermal Analysis): temperature, DSC (Differential Scanning-Calorimetry): temperature, DIL (Dilatometry): length, TMA (Thermo Mechanical Analysis): length (with strain), DMA (Dynamic-Mechanical Analysis): length (dynamic), Dielectric Analysis): Thermo-Microscopy: image, Scanning-Calorimetry): temperature, DEA (Dielectric Analysis): conductivity, Thermo Microscopy: image, combined methods.

Shape memory alloys are special materials which in last decade exhibit a new unexploited property, damping capacity, with many applications in civil construction field.

The SMA energy dissipation devices have been seen in the forms of braces for framed structures /2/, dampers for cable-stayed bridges or simply supported bridges, connection elements for columns and retrofitting devices for historic buildings. Experiments or simulations or both have been carried out to explore the potentials of the SMA-based energy dissipation devices in passive structure control. That research focused on three aspects: modelling for dynamic response of the structures with SMA devices, experimentally verifying the feasibility of the SMA devices and optimizing the SMA devices' design in terms of vibration suppression using experimental and numerical methods.

Several different scale prototypes of the devices were designed, implemented and tested. They showed that the proposed devices have characteristics of great versatility, simplicity of functioning mechanism, self-centering capability, high stiffness for small displacements and good energy dissipation capability. In the work by Han et al. /3/, eight damper devices made of the SMA wires and steel wires were diagonally installed in a two-story steel frame structure, as shown in Figure 1.

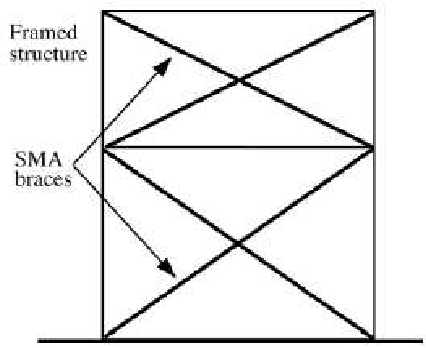

Figure 1 Schematic of the SMA braces for a two-story steel frame /3/

Both experimental analysis and numerical verification were conducted to demonstrate the effectiveness of the SMA dampers on vibration reduction. Experimental comparisons of the frame responses with and without dampers showed that the vibration of the controlled frame decayed very much faster than that of the uncontrolled frame. The simulation has demonstrated that the largest displacement of the controlled frame is only 15% of that of the uncontrolled case /3/.

2. Equipment used in this study

To characterize a metallic material by thermal point of view we use three performing equipments, which investigate the behavior of a shape memory alloy based on copper. Chemical composition was determined through spectrometry analysis using Foundry Master equipment and the microstructure with a scanning electron microscope (SEM) by Vega Tescan brand.

To investigate the behavior of a material during heating with respect for his physical dimension we use a dilatometer equipment type DIL 402, the measuring system employs two high resolution inductive displacement transducers; with its design using low-expansion invar and broad thermostatic control for highest accuracy, reproducibility and long term stability, it is capable of application temperatures up to 1600°C, the horizontal instrument construction offers specific advantages, especially for the dual sample arrangement: homogeneous heating of both samples, simple sample insertion, safety during sample decomposition or melting, and effective protection of the measuring system by gas flow. The operation remains simple and safe through exact sample arrangement at the specific sample support in the cutout of the tube sample support, the individual motorized movement and zero setting of both push rods and the easy to move furnace.

For calorimetric characterization we use a scanning differential apparatus, the DSC 404 Pegasus is part of the economical NETZSCH F3-product line, which is specially tailored to the requirements of comparative material characterization and quality control. The DSC 404 F3 Pegasus can be operated from -150°C to 2000°C with various sensors that are easily exchangeable by the user and various furnace types. The sample chamber can be purged with inert or oxidizing gases in order to remove gases evolved from the sample. The measuring system is vacuum tight (10-2mbar).

The versatility of the DMA 242 C is particularly clear from the variety of sample holders. Depending on the type and consistency of your sample, you can investigate the viscous-elastic properties accurately over a wide modulus range. In addition to the common types of deformation, i.e. 3-point bending, single and dual cantilever bending, compression/penetration, shearing and tension, we also have a series of special sample holders available: e.g. for extremely stiff composite materials and metals or for combination with a dielectric analyzer (DEA) for curing studies. A stainless steel container facilitates measurements in various media (immersion tests). With the use of a thermostat, it is also possible to carry out reliable extended time tests at higher temperatures. The DMA 242 C operates in the broad temperature range of -170 to 600°C. The low-temperature range is achieved with the proven, low-consumption CC 200 L liquid nitrogen cooling system.

The minimal temperature gradient over a sample length of up to 60 mm in the bending mode is unique. An intelligent purge gas system provides for a defined sample atmosphere and protects the measurement electronics from any gases evolving from the sample as well. Frequencies from 0.01 to 100 Hz can be selected and combined with defined stress of up to 16 N and deformation amplitudes of between 0.1 and 240 μm. The digital filtering via Fourier analysis yields an excellent signal-to-noise ratio, which means that even the smallest tanδ values can be resolved. Comprehensive, multi-dimensional calibration of the DMA system guarantees reproducible test results for the stiffness and damping (viscous-elastic) behavior of your sample.

3. Experimental results

A shape memory alloy from copper-zinc-aluminium system was obtain by melting classical method and his chemical composition determined with a spark spectrometer type Foundry Master (using two sparks around 1mm each for an average value of composition) is presented in table 1.

Table 1 Chemical composition of investigated shape memory alloy

Alloy Cu-Zn-Al	Cu	Zn	Al	Pb	Sn	Fe	Ni	Si	Co
1	55	25.4	6.72	2.16	1.18	4.97	0.893	0.684	1.04
2	54.8	27.7	6.16	1.93	1.10	4.19	0.818	0.542	0.866
Average	54.9	26.5	6.44	2.04	1.14	4.58	0.856	0.613	0.953

The alloy $Cu_{54.9}Zn_{26.5}Al_{6.44}$ present a high percent of other elements in composition, like Pb-2.04%, Sn-1.14%, Fe-4.58% and Co-0.953% which modify the thermal alloy characteristics especially the dynamic mechanic behaviour improving the damping capacity of the material.

In figure 2 is presented the alloy microstructure realize with a scanning electron microscope (SEM).

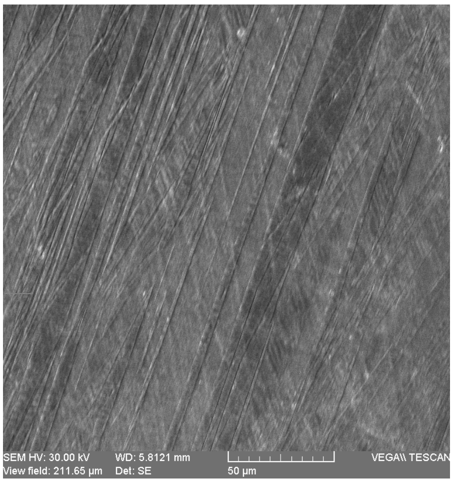

Figure 2 Microstructure of shape memory alloy $Cu_{54.9}Zn_{26.5}Al_{6.44}$

The microstructure of alloy was made on 220x220 µm area with evidence of martensitic variants, characteristic of shape memory alloy. The image is made with a SE (secondary electron) detector for working distance of 16 mm.

The only test that can prove the memory effect of an alloy is dilatometry and for this reason we start with this investigated method.

Sample is on cylindrical form with diameter of 25.620 mm with perfect parallel ends with a temperature range between 32 and 600 °C domain of temperature complete for a nice characterization by the dimensions change with temperature point of view for this kind of alloy.

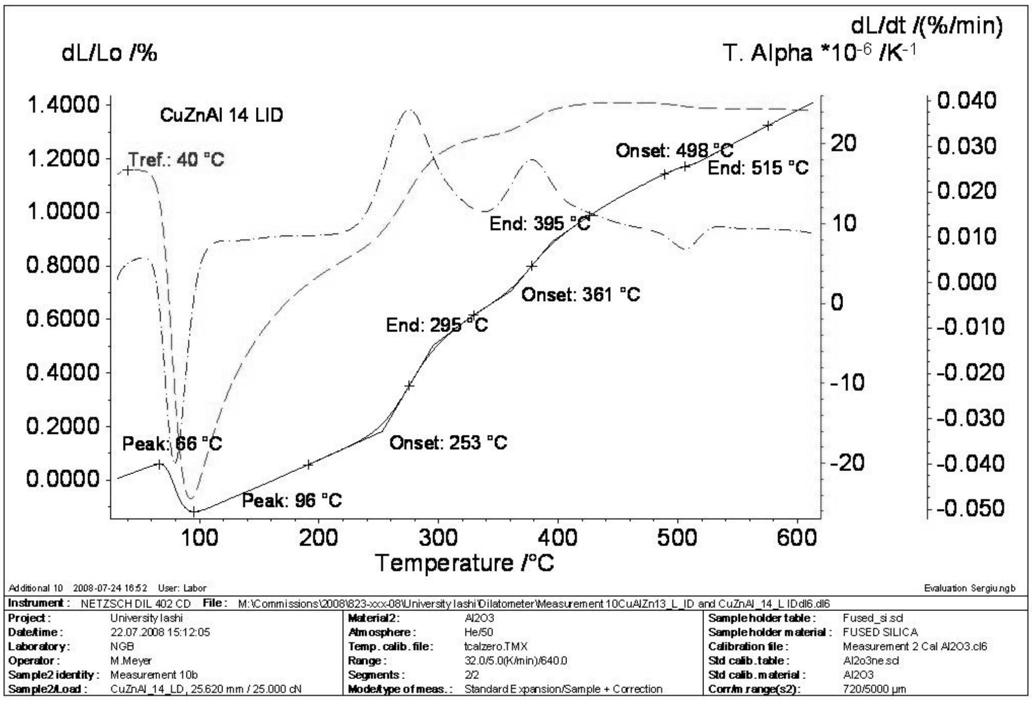

Figure 3 Specific dilatogram of a shape memory alloy $Cu_{54.9}Zn_{26.5}Al_{6.44}$/4/

The dilatogram result is a specific one characteristic for materials with memory effect. After a normal increasing of dimension, with a peak at 66 °C, with temperature the material start to reduce his dimensions with almost 20 µm at 96 °C. In this temperature range the alloy have the transformation points which govern the memory effect. After this temperature field the material behaves normally until 600 °C when the test stops. In figure 4 is presented a DSC analyze made on investigated alloy **$Cu_{54.9}Zn_{26.5}Al_{6.44}$,** test realize in 0-200°C temperature range. The calorimetric test show all the phase transformation that take place in alloy, and as can be seen from figure 4 this transformations are in 75-125 °C range, confirming the dilatometry test.

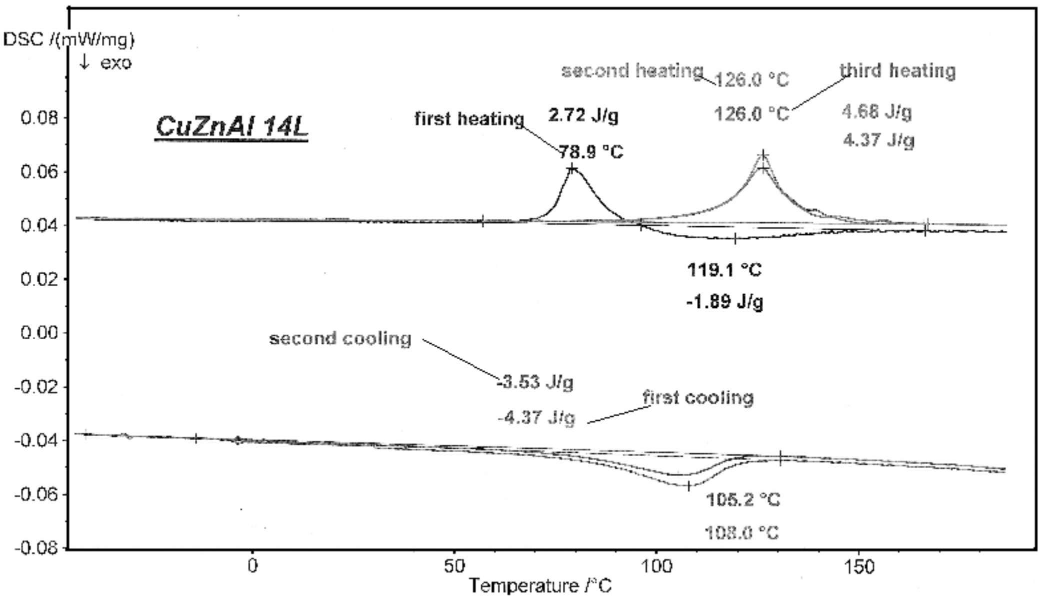

Figure 4 DSC analyze of a $Cu_{54.9}Zn_{26.5}Al_{6.44}$ shape memory alloy/4/

Generally the endothermic peak's reveal at heating correspond to martensite reversion and the exothermal peak's appear at cooling, correspond to direct martensite transformation. /4/ Using a DSC diagram it is easy to determine the transformation points at heating and cooling process of the alloy. Between one and more heating cycles can appear differences on transformation point's values until the alloy suffer a thermal stabilization.

The next test was realize with a dynamic mechanical analyzer, the alloy $Cu_{54.9}Zn_{26.5}Al_{6.44}$ mechanically prepared with dimensions 20x7,95x0,55 mm and as test parameters heaving heating regime were from 0,5 to 0,5 °C, at work frequency 1 Hz and range temperature from 30to 300 ºC temperature.

According to ISO 6721-1 /5/, the storage modulus E´ represents the stiffness of a viscoelastic material and is proportional to the energy stored during a loading cycle. It is roughly equal to the elastic modulus for a single, rapid stress at low load and reversible deformation, and is thus largely equivalent to the tabulated figures quoted in DIN 53457. In the same ISO standard, the loss modulus E´´ is defined as being proportional to the energy dissipated during one loading cycle. It represents, for example, energy lost as heat, and is a measure of vibrational energy that has been converted during vibration and that cannot be recovered. According to /5/, modulus values are expressed in MPa, but N/mm^2 is sometimes used. The real part of the modulus may be used for assessing the elastic properties, and the imaginary part for the viscous properties /6/. The phase angle δ is the phase difference between the dynamic stress and the dynamic strain in a viscoelastic material subjected to a sinusoidal oscillation. The phase angle is

expressed in radians (rad) /5/. The loss factor tan δ is the ratio of loss modulus to storage modulus /5/. It is a measure of the energy lost, expressed in terms of the recoverable energy, and represents mechanical damping or internal friction in a viscoelastic system. The loss factor tan δ is expressed as a dimensionless number. A high tan δ value is indicative of a material that has a high, nonelastic strain component, while a low value indicates one that is more elastic.

Dynamic elasticity modulus E' decrease from a value of 67000 MPa at room temperature until a value of 47554.41 MPa at 89 °C, minimal value corresponding to maximal peak of internal friction and after this increasing until a value of 78 000 MPa at 300 °C, can be observe as in CuMnAl alloy case, the link between the maximal value of internal friction and the minimum value of elasticity modulus. So for this material the internal friction rich quite a big value of 0,11635 for a temperature of 89.73028 °C. This peak is quite considerable near to practical application point and can be improve by a cold plastic deformation or a proper heat treatment or different classical methods like alloying with small amounts of elements like iron, tantalum, magnesium or molybdenum. Corresponding to this value of internal friction we have a elasticity modulus with value of 47556.07277 MPa /7/.

After reaching this peak the internal friction decrease progressively until temperature of 300 °C another peak until this temperature was not seeing. The high value of internal friction makes from this alloy a good solution for amortization elements if we control the alloy temperature. The temperature were IF is maximum is a approachable value, being easy to heat the material until 90 ºC./7/

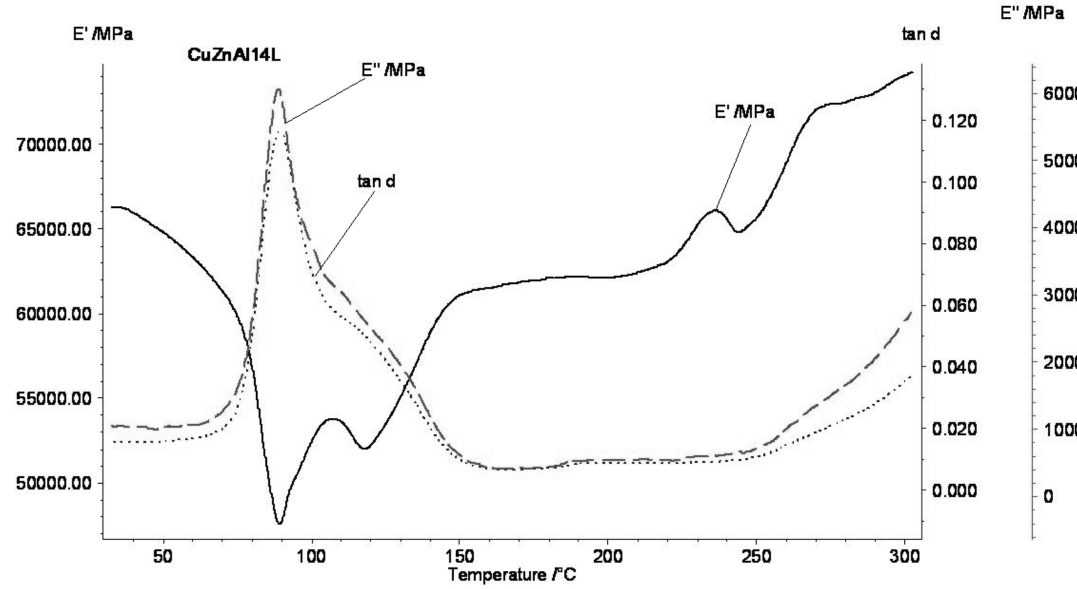

Figure 5.Variation diagram of internal friction and elasticity modulus with temperature of a shape memory alloy type $Cu_{68,1}$ $Zn_{13,2}$ $Al_{4,85}$/4/

E'' "loss modulus" represent an amortization term describing the energy dissipation capacity in heat when a material is deform and appear named imaginary modulus as a part of complex elasticity modulus, E=E'+E'.

4. Conclusions

- An alloy from copper-zinc system was obtaining in classical conditions, and after investigation with a differential dilatometer reveals nice shape memory effect.
- Using combined equipments we can analyze and conclude on thermal behavior at heating and cooling of a shape memory alloy like transformation points, damping capacity and elastic modulus.
- Small value of internal friction at room temperature of a shape memory alloy can't be used in energy dissipation applications. Contrarily the same Cu-Zn-Al SMA presents a nice peak of internal friction around temperature of 90 ºC, in transformation points range, of 0,116 which can be use for practical applications.

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CARACTERIZAREA UNUI ALIAJ CU MEMORIA FORMEI PRIN ANALIZE TERMICE

Abstract: Un aliaj cu memoria formei tip Cu-Zn-Al a fost analizat cu diferite echipamente pentru caracterizarea termica a acestuia. Aliajul a fost cercetat cu un microscop cu scanare de electroni pentru determinarea microstructurii si apoi cu echipamente ca dilatometrul diferential (DIL) pentru stabilirea prezentei efectului de memoria formei cu un calorimetru cu scanare diferentiala pentru determinarea punctelor de transformare si cu un analizor mecanic dinamic (DMA) pentru determinarea unor proprietati mecanice.