PROCESSING SMART WIRES FROM CU-AL-NI SYSTEM

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ABSTRACT

In this paper we present some results concerning plastic deformation applied to shape memory alloy and two most direct characterization methods for shape memory. We work with a copper based alloy- Cu Al13Ni 4. Over eight percent in aluminum the plastic deformation on the copper aluminum alloys is very difficult to do. Our purpose was to obtain the thin wires. The first material shape was cast ingots very fragile. We choose to work with a direct extrusion method because the high fragility of this material required a three dimensional stress compression scheme.

KEYWORDS: smart memory alloy, extrusion

1. Introduction

The motivation for this research is the market requirement for a new materials class Shape Memory Alloy (SMA). This materials are expensive because use the expense elements also use expense technologies. In these conditions our purpose was to choose an easy obtain smart material and to find adequate processing. We chose the copper based SMA using the former team experience in cooper alloys [1], [2], [5].

Shape Memory Alloys is a designation that is applied to that group of metallic materials that demonstrate the ability to return to some previously defined shape or size when subjected to the appropriate thermal procedure. Generally, these materials can be plastically deformed at some relatively low temperature, and upon exposure to some higher temperature will return to their shape prior to the deformation.

A shape memory alloy may be further defined as one that yields a thermoelastic martensite.

In this case, the alloy undergoes a martensitic transformation of a type that allows the alloy to be

deformed by a twinning mechanism below the transformation temperature.

The deformation is then reversed when the twinned structure reverts upon heating to the parent phase. [6]

2. Melting conditions

The melting process has been developed in a tipping furnace graphite melting pot. The maximum charge weight: 100 kg. For the charge composition we used electrolytic copper plates, nickel and titanium plates.

Practically in burning melting pot was introduced protection material. When this material came incandescent the electrolytic cooper preheated at 200 ^oC was introduced.

The method supposes ingot casting followed melting to avoid pre alloys for purity reasons. At same time we had a special attention for melting bath protection. In this way we used $Na_2B_4O_7$ and NaCl. Deoxidation was made with CuP_{10} and degasification with Cl_2Mn .

The melting scheme is:



The melting conditions were managed so that cooper fusion to be faster. Final temperature was 1150° C. In this moment was made a first deoxidation with CuP₁₀. Nickel also pre heated was added in small parts. In this time a bath agitation was made. From initial weight 10% was retained and is used in this moment for reduce the bath temperature.

This reduce is necessary for aluminum addition. The second deoxidation followed.

The chemical composition was determined using a Qantovac device based on the optical spectral analysis. The result is presented in table 1.

The structure for this alloy (fig.1) and a classic bronze microstructure are very different.

alloy 1									
Nr.crt	Sn	Pb	Zn	Mn	Fe	Ni	Si	Al	Cu
4.1	0.02	0.01	0.79	0.09	0.27	4.00	0.04	12.88	Rest



Fig.1. Alloy with 13%Al and 4% Ni, Alloy: Cast aluminium bronze Chemical composition: 13 %Al, 4 Ni, rest Cu Magnification: 50x



Fig.2. Stress scheme direct extrusion

3. Plastic Deformation

Before the plastic deformation technology was choose necessary behavior plastic deformation study was made [1], [2], [4]. All those study confirm that upper 10% Al plastic properties of alloy are pronounced decrease.

In these conditions direct hot extrusion process was designed. This deformation process must resolve problem to obtain cylindrical shapes 10 mm diameter and 120 mm length starting from normal conic molded ingot (Φ 25- Φ 40)x 150 mm, aluminum bronze 4%Ni.



Fig.3. Direct extrusion die

The direct extrusion is only plastic deformation method which assures tri dimensional compressive stress (fig. 2) indicates for such hard deformable alloy.

In this sense was designed a direct extrusion dies (fig. 3) to assure plastic deformation from 35 mm diameter to 3mm diameter wires (fig. 4). The 13 % Al extruded semi products microstructure show a martensitic aspect (fig. 5).



Fig.4. A- ingot, B- 20 mm extruded shape, C-10 mm extruded shape



Smart memory alloy deformation degree 111% Chemical composition 12.88%Al, 3.84% Ni, rest Cu Microstructure: γ_1 ' Magnification: 100x



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Fig. 5. Microstructure 13% Al smart memory alloy

The last plastic deformation was made from 10 mm diameter to 3 mm wires.

In figure 6 is presented the semi product and the extrusion dies.



Fig.6. Extrusion die, punch, 3 mm extruded wire



Fig.7. Extruded SMA wires

Our next step study has been concentrated on aged and quenched specimens of Cu-Al-Ni alloy, solubilized and we performed, the Differential Scanning Calorimetry and Electrical Resistivity characterization techniques. The specimens were solubilized in austenitic domain at 850° C for 30 minutes. Homogenized specimens were immediately quenched in iced water in order to obtain β -type martensite.

After these treatments the calorimetric experiments were performed by means of SETARAM 92 instrument at a rate of 10°C per minute, between - 50°C and 250°C. The cooling treatments were acted by using liquid nitrogen. The electrical resistivity to identify the crystalline phases present in materials and to measure the structure properties was made with a THERMO ELECTRON COOP HAAKE DC50-K40

4. Characterization methods

There are two major methods of characterizing the transformation in SMAs and a large number of minor methods:

Differential Scanning Calorimetry

Electrical resistivity

The objective of the present work is to analyze the variation of the temperature with thermal cycling using the Differential Scanning Calorimetry (DSC), and the electrical resistivity to identify the crystalline phases present in materials and to measure the structure properties.

The most direct method is by DSC. This technique measures the heat absorbed or given off by a small sample of the material as it is heated and cooled through the transformation temperature range. The sample can be very small, such as a few milligrams, and because the sample is unstressed, this is not a factor in the measurement. The endotherm and exotherm peaks as the sample absorbs or gives off energy due to the transformation, are easily measured for the beginning peak, and end of the phase change in each direction.



Fig.8. DSC SETARAM 92

The second method often used is to measure the electrical resistivity of the sample as it is heated and cooled. The alloys exhibit interesting changes and peaks in the resistivity (by up to 20%) over the transformation temperature range; however, correlating these changes with measured phase changes or mechanical properties has not always been very successful. Also, there are often large changes in the resistivity curves after cycling samples through the transformation a number of times. Thus, resistivity is often measured as a phenomenon in its own right, but is rarely used to definitely characterize one alloy versus another.

The technical importance of most engineering materials is based on their mechanical, electrical or magnetic properties, which should, normally, be as independent as possible from environmental influences.

5. Experimental results DSC analysis

A sample weighting 0.0347g was cut from a Cu-Al-Ni wire, etched with a solution of 1:1 HNO₃ and H₂O for 13 minutes to remove the layers affected by the cutting. The calorimetric experiments were performed between -50°C and 250°C at a heating rate of 10°C/min. The cooling treatments were carried out by using liquid nitrogen.



Fig.9.

The curve from (fig 9) was a closed one with no phase transformation, with no peak on the reverse martensite transformation, between 73°C and 159°C present a large peak.

The quenched curves (fig.10) showed a phase transformation both on forward and reverse direct martensitic transformation.



Fig.10. DSC curves for a solubilized alloy at 850°C

6. ER analyses

We can observe a straight line, the resistivity is increasing with the temperature rising, showing that there is no phase transformation at the 9 months aged at room temperature specimen.



Fig11. ER aged sample

At 850°C solubilised sample there is a phase transformation that starts around 85°C, the resistivity starts to decrease until 103°C when the phase is finished and the resistivity has the tendency to increase. On cooling a reverse phase transformation takes place.



Fig.12. ER quenched sample

In table 2 we have transformation temperatures obtained from DSC and ER measurements.

Table 2 Solubilized at Transformation 850°C temperatures DSC ER 80 As 92 138 103 Af 152 90 Ms Mf 84 60

7. Conclusions

The extruded wires have a martensitic structure to the normal temperature. Dimensions and micro structural aspect allow continuing to the next step establishing thermo mechanical treatments to induce the shape memory effect. The Cu-Al-Ni, after solubilization followed by quenching, showed the phase transformations that are typical of the Shape Memory Effect (by DSC and Electrical Resistivity).

Acknowledgement

The authors would like to acknowledge the support from CENIMAT FCT/UNL Caparica, Portugal, specially Mister Professor F.M. Braz Fernandes and his team.

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