STUDENT PAPER

PREPARATION AND CHARACTERIZATION OF LOW COST Cu-Al-Be SHAPE MEMORY ALLOY FOR AEROSPACE APPLICATIONS

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Abstract

Recently it has been found that a small addition of Beryllium (Be) widens the transformation temperature in Cu-Al based Shape Memory Alloys (SMA), that is particularly interesting in the industrial applications point of view. In this paper, the processing of Cu-Al-Be SMA by gravity die casting technology was described. The specially designed heat treatment procedure was designed to stabilize the SMA effect in this alloy. The DSC curve showed that this alloy has the transformation temperature in the range of 65°C to 120°C, and also exhibits the hysteresis behavior. Microstructure examinations confirmed the presence of lath martensite phases at room temperature. The bend test was used to prove the existence of the shape memory effect in this alloy.

***Keywords**: Cu-Al-Be Shape Memory Alloy; Gravity Die Casting; DSC Curve; Bend Testing; Martensite*

Introduction

Shape Memory Alloys (SMA) are the unique class of smart metallic materials which have an intrinsic ability to recover the predefined shape upon appropriate thermal and/or mechanical treatment without any residual strain. The recovery of the strain with the thermal (one way shape memory effect) or mechanical treatment (pseudo elasticity) is attributed to the thermo elastic martensitic transformation [1]. The thermo elastic martensite phase formed in this alloy is soft and has many variants. This phase is formed by a diffusionless solid state shear like mechanism called twinning. The martensite variants have the same crystal structure, but differ from orientation, and formed from a single austenite crystal. Upon loading, these twinned martensite variants accommodate large recoverable strains upto the point where all twinned variants in the structure become one single variant, beyond which martensite deforms plastically. When the alloy is heated above the transformation temperature, shape change results from phase transformation of martensite to austenite accompanied by change in crystal structure called one way shape memory effect [2]. These properties make shape memory alloys research most technologically important since the last few decades.

Nitinols (Ni-Ti shape memory alloys) is one of the classical examples unraveling the commercial potential of shape memory alloy applicability. The high susceptibility to pick up of gaseous elements mainly oxygen, solidification defects such as pipes, shrinkage, segregation, inclusion etc., and a criterion of maintaining equiatomic proportion of Ni and Ti in the alloy to exhibit shape memory behavior demand for costlier vacuum induction melting/vacuum arc remelting technique to have rigorous control over alloy compositions. Therefore, Ni-Ti alloys are quite expensive, which restricts their applications to niche markets such as medical stents, industrial automation, aerospace and defence.

Recently it is found that Cu based alloys e.g., Cu-Al-Zn, Cu-Al-Mn, Cu-Al-Be also exhibit shape memory effects (SME). Cu based SMAs have been preferred since they have good memory properties, and most importantly, low production cost [3, 4, 5]. In these alloys, the SME is achieved through a thermo elastic martensitic transformation. In Cu-Al alloy, the disordered BCC, β, austenite phase, which is responsible for shape memory behavior, is stable at high temperatures [6]. The small addition of Be into Cu-Al alloy brings down the martensite transforma-

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tion temperature without affecting the β phase stability, and also favors the DO_3 ordered structure in the metastable austenite [7]. Veloso et. al [8] and Belkahla et. al [9] reported that the addition of only 0.1 wt % of Be reduced the phase transformation temperature of this alloy by approximately 100°C. When the alloy is rapidly cooled from the disordered β phase to suppress equilibrium phases, α (FCC structure), and v_2 (Cu_9Al_4), the retained metastable β phase becomes the ordered DO_3 austenite structure before transforming to the thermo elastic martensite [10, 11, 12]. This phase transformation imparts the shape memory behavior in this alloy [13]. The significant drop of the transformation temperature with a small amount of Be addition makes a Cu-Al-Be SMA scientifically interesting and technologically important. In addition, the processing of this alloy is relatively simple. It can be casted in the air that makes the processing cost inexpensive. Besides, this alloy has many interesting properties e.g., superelasticity, an excellent capacity to absorb sound, vibration and mechanical waves, high mechanical strength, resistance to corrosion etc., [14]. In the present study, an attempt was made to cast Cu-Al-Be SMA by the gravity die casting technology. The alloy was characterized to identify the transformation temperature using the differential scanning calorimeter (DSC) and the phases using the optical microscope. The one way shape memory effect of this alloy was also verified by bend test.

The paper was organized as follows: the processing, the heat treatment and testing aspects of the Cu-Al-Be SMA described in detail in the next section. The results of transformation temperatures, bend test results to understand one way shape memory ability of the alloy were discussed in the subsequent section. Additionally, microstructures of the Cu-Al-Be SMA were presented. Finally, conclusions were drawn based on the results.

Materials and Methods

Materials and Processing

The alloy used in this study composed of Cu-11.7 wt % Al-0.47 wt % Be eutectoid alloy system. Copper, aluminum and beryllium metal sheets (Purity ~ 99%) were procured from Metalex India Pvt. Ltd, Bangalore. Sheets were air melted in a graphite crucible by induction heating in a VAP induction furnace (15KW/10KHz/400V). The melt was poured into preheated grey cast iron die mould of dimensions 150 mm x 100 mm x 5 mm (length x width x thickness) for strip casts and 8 mm x 30 mm (diameter x height) for cylindrical casts and allowed to solidify under gravity pressure in the air atmosphere by the gravity die casting technique. The gravity die casting setup and the induction furnace used in this study are shown in Figs.1-2 respectively. The casts were homogenized at 900°C for 6 h in an electric resistance furnace to avoid any micro segregation and dissolve the dendrites. Then the casts were hot rolled into 1 mm thick sheet at 900°C in a two-high roll mill. The process chart of the Cu-Al-Be alloy preparation is given in Fig.3.

Heat Treatment

The heat treatment cycle followed for this alloy is given in Fig.4. To form a continuous β-phase (austenite) in the microstructure, samples were given betatization heat treatment. In this treatment, samples were heated to 900°C and held for 30 min. The longer holding time may cause severe grain growth which has an adverse effect on the shape memory characteristics of this alloy. Subsequently, samples were subjected to step quenching in two stages, initially in a hot water bath (100°C), followed by cold water (28°C). This treatment was essential for this alloy to exhibit shape memory behavior because the high temperature homogenization and the quenching rate were the main factors deciding the reverse phase transformation (shape memory behavior) in this alloy [15]. In addition, the step quenching alleviates the problems of quench cracks. It is also reported that martensite stabilization occurs during the step quenching by vacancy pinning mechanisms [16]. After heat treatment, samples were ground to remove oxide scales before testing.

Testing

Differential Scanning Calorimeter (DSC)

Strips of 3 mm x 1 mm (length x thickness) were cut from the sheet using a low speed diamond saw for DSC experiments. DSC analysis of samples was carried out in a Setaram DSC823 calorimeter under the nitrogen gas flow. The critical parameter in the DSC analysis of SMAs is the choice of heating/cooling rate. The choice of high heating rates often decreases the resolution of the adjacent peaks swhereas, at very low heating rates, the sensitivity decreases and in extreme cases, the peaks may completely disappear. Further, the high cooling rates make the detection of M_s and M_f temperatures impossible due to the reduced response time of the instrument. Hence the proper choice of heating/cooling rate is a prerequisite for accurate prediction of phase transformation temperatures. The temperature range of 30°C - 180°C was chosen as an optimum range to conduct the test after the number of preliminary trials. The heating and cooling rates were optimized to +20°C/min and -10°C/min, respectively. The transformation temperature of phases was identified from the DSC curve.

Microstructure Examination

Samples were cut using a low speed diamond saw for microstructure examinations. These were mechanically polished on emery sheets of 200-2000 grid sizes followed by cloth polishing with 0.1 mm alumina paste to get very fine polished surface. Samples were etched in an etchant solution of 2 g $K_2Cr_2O_7$ - 8 ml H_2SO_4 - 2 ml HCl -100 ml H_2O. These samples were examined under an upright optical microscope (Olympus - Japan).

Bend Test

Bend test was carried out to examine the shape memory behavior of this alloy. Strips with dimensions of 50mm x 4 mm x 1 mm (length x width x thickness) were machined from the heat treated sheet. The procedure of bend test and the schematic diagram of bend test (as shown in Fig.5) were given in Feng and coworkers [17]. These samples were bent in a circular shape mandrel to form a round shape at room temperature. It was unloaded and allowed to spring back. After the spring back, the angle formed by the sample, θ_s, was measured by drawing the projection of the sample shape in the white paper and measuring the angle using protractor. Subsequently, the sample was heated to (above A_f temperature), the shape change by the shape memory effect was evaluated by measuring the angle, θ_t. The surface strain after bending, ε, the shape memory strain, ε_{SME}, and the recovery ratio, R, were evaluated based on the equations (1-3) given by Feng and coworkers [17]. The shape memory effect, % SME was calculated based on the Eq.(4).

$$\varepsilon = \frac{1}{t+D} \tag{1}$$

$$\varepsilon_{SME} = \frac{(\theta_t - \theta_s)\,\varepsilon}{180^o} \tag{2}$$

$$\%\ SME = \frac{\theta_m}{(180^o - \theta_e)} \tag{3}$$

$$R = \frac{(180^o - \theta_t)}{180^o} \tag{4}$$

Where t and D are the specimen thickness and the diameter of a mandrel roll respectively.

Results and Discussion

Transformation Temperature

The DSC curve of the Cu-Al-Be SMA is presented in Fig.6. The endothermic peak and the exothermic peak in Fig.6 correspond to martensite to austenite transformation and austenite to martensite transformation respectively. Both the peaks showed that this alloy undergoes transformation from martensite phase to austenite phase or vice versa at a well defined temperature. The reverse transformation shows the existence of the shape memory effect in this alloy. In addition, it also implies that the heat treatment cycle designed is capable to stabilize the martensite phase at room temperature. The sound shape memory characteristics of this alloy were attributed to greater height and sharpness of the peak [1], as evident in Fig.6. From this curve, A_s, A_f, A_{max}, M_s, M_f and M_{max} temperatures were determined as 95°C, 140°C, 120°C, 45°C, 90°C and 65°C respectively. A_s, A_f, A_{max}, M_s, M_f and M_{max} denote the austenite start temperature, the austenite finish temperature, the austenite maximum phase transformation rate temperature, the martensite start temperature, the martensite finish temperature, and the martensite maximum phase transformation rate temperature respectively. The observed change in temperature between M_s and A_f , M_f and A_s indicates that the alloy exhibits the hysteresis behavior. The hysteresis characteristics impart the high damping capacity from energy dissipation through hysteresis cycles of reverse transformation.

Microstructure

The microstructures of the room temperature phase (martensite) and high temperature phase (austenite) of the Cu-Al-Be SMA are shown in Figs.7-8. Long martensite plates with twin bands were observed in the microstructure of the martensite whereas the clear equiaxed grains are seen in the austenite phase. Balo and coworkers [18] have studied the crystal structure of the same alloy using x-ray diffraction. They have reported that the austenite phase has a DO_3 super lattice structure and the martensite phase has a 18R structure. As precipitates formation was mainly responsible for the shape memory effect deterioration [7,19], their absence in the martensitic matrix confirmed the good shape memory characteristics of the alloy fabricated by us.

Shape Memory Effect

Saburi and coworkers [20, 21] have reported the formation of 24 possible martensite variants of the same crystal structure with different orientation after rapid cooling to room temperature from 900°C. During bend test, the application of force deforms the martensite variants which are oriented favorably to the loading axis. These variants grow at the expense of unfavorably oriented variants and accommodate large strains by self-accommodation mechanism until all variants become single variant. Beyond which, martensite deforms plastically. Subsequently, heating of bent sheets above A_f regains in the original shape before bend deformation by phase transformation. The calculated parameters from the bend test results based on Eqs.1-3 are given in Table-1. The surface strain of bending deformation, ε, the shape memory strain, ε_{SME}, the recovery ratio, R, and % SME were 3.03%, 1.26%, 0.33 and 88.88% respectively.

Conclusions

The Cu-Al-Be shape memory alloy was processed successfully through the gravity die casting method. The material characterization results are as follows:

- DSC experiments of the Cu-Al-Be SMA show that the A_s, A_f and A_{max} were found to be 95°C, 140°C and 120°C from the exothermic peaks and M_s, M_f and M_{max} were found to be 45°C, 90°C and 65°C from the endothermic peaks. The presence of reverse transformation confirmed the existence of shape memory effect.
- The observed hysteresis behavior from a Cu-Al-Be SMA shows its high damping capacity potential.
- Microstructual studies of the Cu-Al-Be SMA reveal the phase of long martensite plates with twinned regions at room temperature and equiaxed austenite grains at high temperature (A_f).

Table-1 : Bend Test Results of the Cu-Al-Be Alloy (Measured Values of Angles, θ_t and θ_s, are 120° and 45° respectively)

Surface Strain (ε)	Shape Memory Strain (ε_{SME})	Recovery Ratio (*R*)	% Shape Memory Effect
3.03 %	1.26 %	0.333	88.88 %

- The bend test results showed that the surface strain after bending deformation, ε, the shape memory strain, ε_{SME}, the recovery ratio, R, and % SME were 3.03%, 1.26%, 0.333 and 88.88% respectively and also confirmed the shape memory behavior of the Cu-Al-Be SMA.
- Bend test, microstructure and DSC studies confirmed that the chosen heat treatment cycle was appropriate to induce good shape memory characteristics in the Cu-Al-Be SMA.

From the above results, it can be proposed that Cu-Al-Be alloys may be very well employed in room temperature smart system applications due to its low transformation temperature range, and low cost processing.

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Fig.1 Gravity Die Casting of the Cu-Al-Be Alloy

Fig.2 Induction Furnace Setup (15KW/10KHz/400V)

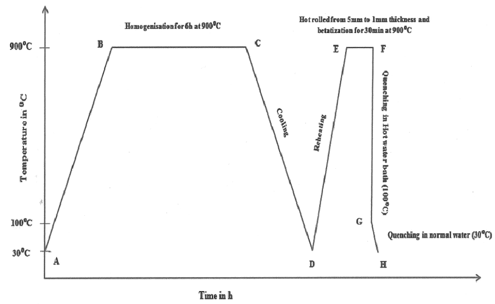

Fig.4 Heat Treatment Cycle

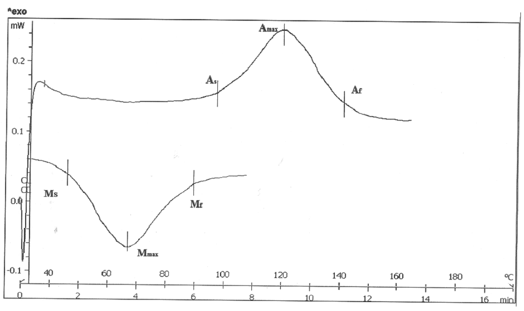

Fig.6 DSC Curve of the Cu-Al-Be Alloy Showing Phase Transformation Temperatures

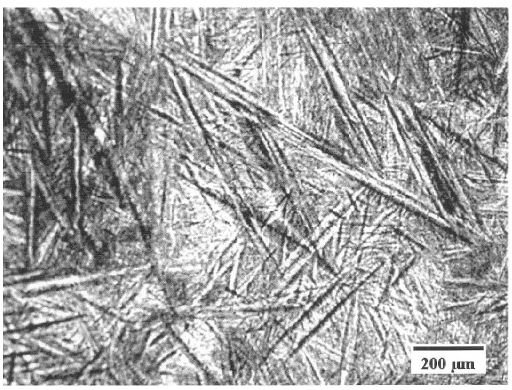

Fig.8 Microstructure of the Lath Martensite in the Cu-Al-Be Alloy

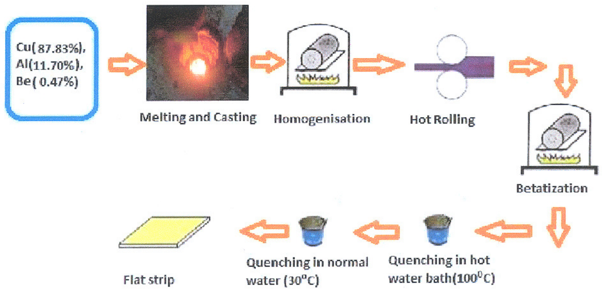

Fig.3 Process Flow Chart of the Cu-Al-Be Aloy

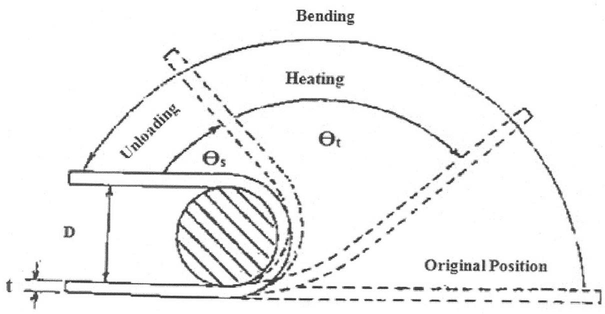

Fig.5 Schematic Diagram of Bend Test. D, t, θ_s and θ_t are the mandrel roll diameter, the sheet thickness, the angle formed after spring back of the sheet after bending at room temperature and the angle formed on heating to above A_f temperature

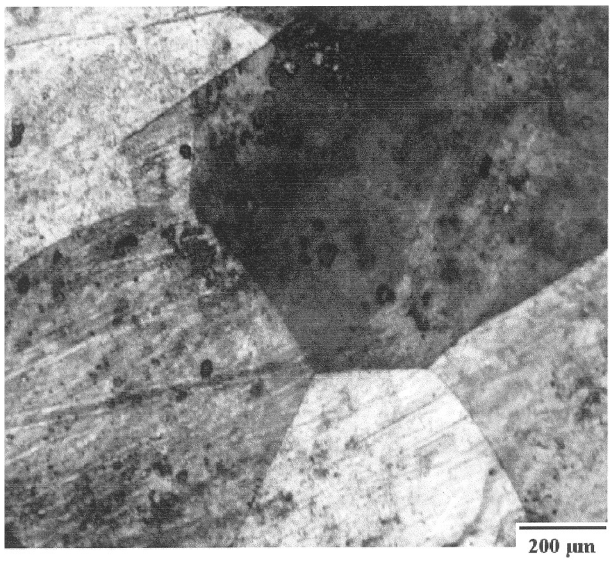

Fig.7 Microstructure of the Equiaxed Grain Austenite in the Cu-Al-Be Alloy