www.sphinxsai.com

ChemTech

International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN : 0974-4290
Vol.6, No.3, pp 1715-1717, May-June 2014

ICMCT-2014 [10th – 12th March 2014]
International Conference on Materials and Characterization Techniques

Characterization of mechano-chemically synthesized Cu-Al-Mn shape memory alloy powders and spark plasma sintered compacts

R. Sai Kishore, R. K. Nandhakumaar, V. Gokuul, A. Siddharthan

Department of Mechanical Engineering, College of Engineering Guindy, Anna University, Chennai, India.

Abstract: Cu-Al-Mn alloy powders were synthesized by mechanical alloying by 9 hours of ball milling of elemental powder mixtures. X-ray diffraction (XRD) analysis of powders milled at different intervals of time was useful in identification of alloy formation, lattice strain and crystallite size. The obtained crystallite size at the end of 18 hours of milling is 9 nm. The individual elemental peaks disappeared and the peak shifts got stabilized before 9 hours of milling, indicating the completion of alloying. Scanning electron microscopic (SEM) images of alloyed powder revealed a typical lamellar structure characteristic of ball milling. Differential Scanning Calorimeter (DSC) analysis of powder indicated the presence of partially unalloyed copper. Spark plasma sintering (SPS) of the alloy powder produced pellet with an average crystallite size of 25 nm.
Keywords: Cu-Al-Mn shape memory alloy powders, spark plasma sintered compacts, Characterization, mechano-chemically synthesis.

Introduction and Experimental:

Among shape memory alloys (SMA), Cu-based alloys are less expensive than the Ni–Ti based alloys and show better shape memory effect (SME) than the Fe-based alloys. However, the bulk polycrystalline Cu-based SMAs such as Cu–Al–Ni and Cu–Zn–Al are too brittle to be sufficiently cold-worked. Particularly Cu-Al-Mn alloys have relatively greater ductility [1]. It is known that even slight variations in alloying elements would strongly influence SME. Mechanical alloying (MA) has advantage of control the alloy composition than by the casting and producing nanostructured materials. *Rezwani et al* [2] reported globular-shaped Cu-12.5%Al-5%Mn alloy nanopowders with average crystallite size of 7 nm after 15 hrs of milling. *Portier et al* [3] carried out Spark Plasma Sintering (SPS) of Cu-Al-Ni powders at 650-700°C with better mechanical properties. *Amini et al* [4] reported that lesser hours of milling produced nanocrystalline Cu solid solution of Cu-11.5%Al-4%Mn while the extent of the amorphous phase increased with milling time.

Mechanical alloying was performed in planetary ball mill (FRITSCH Mono Mill PULVERISETTE). The elemental powders of Cu (-140+325 mesh, 99.5% pure), Al (-150 mesh, 99.8%) and Mn (-40+325 mesh, 99.6% pure) were mixed in the weight ratio 82.5%, 12.5% and 5% respectively as reported elsewhere [2]. The powders were milled with WC balls of 10 mm diameter for 3 to 18 hours in toluene medium (to avoid oxidation) and coded as CAMx (x - milling time in hours). The ball to powder weight ratio (BPR) was 15:1 and milling speed was 300 rpm. The X-ray Diffraction (XRD) analysis of the powders was conducted using PANalytical Diffractometer with Cu K_α radiation in the 2θ range of 20º to 90º and a step size of 0.02º. Scanning

Electron Microscopic (SEM) studies were performed using Carl Zeiss MA15/EVO 18 equipment. Differential Scanning Calorimeter (DSC) study of alloy powders was performed in N_2 atmosphere with a heating rate of 10 ºC/minute upto 1500 ºC using NETZSCH STA 449 F3 Jupiter. Spark plasma sintering (SPS) was carried out using DR. SINTER SPS-625 on the alloyed powders at a maximum temperature and pressure of 700 ºC and 60 MPa respectively.

Results and Discussion:

XRD patterns of powders milled for different number of hours are shown in Fig. 1 and show that the elemental peaks of Al and Mn are not detected after 9 hours of milling. The XRD patterns show an increase in full-width at half-maximum (FWHM) of the peaks indicating reduction in crystallite size and increase in lattice strain with milling time.

The peak corresponding to (1 1 1) plane of Cu at the 2θ position of 43.39º in CAM0 slightly shifts rightwards in CAM3 and CAM6 to 43.59º and 43.932º respectively and can be ascribed to the compressive strain. The compressive strain in the Cu lattice is due to the dissolution of Al and Mn atoms in it. The strain produced by dissolution of Al atom into Cu lattice would be greater due to the atomic radius of Al (143 pm) that is larger that of Cu (128 pm). The steady shift in 2θ position of Cu (1 1 1) peak upto CAM6 indicates the formation of solid-solution of Cu. Since there is no significant peak shifts and no change in lattice strain after 9 hours of milling, a saturated solid solution must have been formed between 6 and 9 hours of milling (Table 1).

The crystallite size and lattice strain were calculated using the following equations.

$$\text{Crystallite Size (average)} = k\lambda / (B_{size} \cos\theta) \quad (1)$$

$$\text{Lattice Strain} = B_{strain} / (4 \tan\theta) \quad (2)$$

where $B_{size} = B_{obs} - B_{std}$, $B_{strain}^2 = B_{obs}^2 - B_{std}^2$, in which B_{obs} is FWHM from the peak profile of the experimental sample, B_{std} is FWHM obtained from that of a standard specimen. In this work, silicon sample ($B_{std} = 0.09$) was used as reference standard to account instrumental broadening. The average crystallite size is 9 nm after 18 hours of milling.

SEM images on the alloyed powders shown in Fig. 2 indicate that the powder particles initially have typical lamellar structure due to the impact of the balls, which would eventually become irregular as the milling time increases.

The DSC study of CAM9 powders (Fig. 3a) revealed two melting peaks at 1038 ºC (melting of alloyed Cu) and 1083 ºC (melting of remnant unalloyed Cu). This remnant Cu was not revealed in XRD because of detection limits. The XRD pattern and the photograph of the CAM9 sintered pellet is shown in Fig. 3b. The average crystallite size in the sintered pellet was 25 nm.

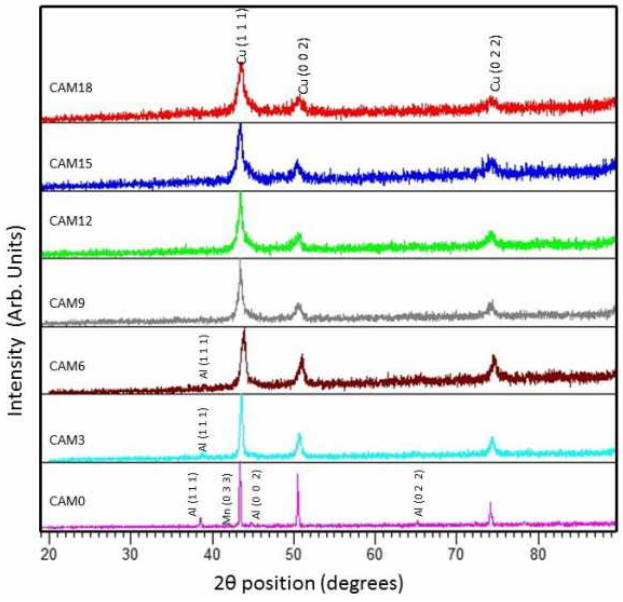


Fig. 1. XRD patterns of as-mixed and milled powders

Table 1. Peak positions and peak shifts in various milled powders

Sample Code	Cu (1 1 1)		Cu (0 0 2)		Cu (0 2 2)		Al (1 1 1)	Mn (0 3 3)	Crystallite Size (nm)	Lattice Strain (x 10^{-3})
	Peak Position	Peak Shift	Peak Position	Peak Shift	Peak Position	Peak Shift	Peak Position	Peak Position		
CAM0	43.38	0	50.5	0	74.17	0	38.52	43.08	376	0.717
CAM3	43.58	0.2	50.7	0.2	74.41	0.24	38.82	-	27	3.54
CAM6	43.91	0.53	51.06	0.56	74.66	0.49	39.07	-	16	5.42
CAM9	43.46	0.08	50.6	0.1	74.28	0.11	-	-	15	6.13
CAM12	43.46	0.08	50.6	0.1	74.3	0.13	-	-	14	6.32
CAM15	43.46	0.08	50.55	0.05	74.28	0.11	-	-	11	7.92
CAM18	43.5	0.12	50.74	0.24	74.43	0.26	-	-	9	8.7
CAM9SINT	43.24	-0.14	50.24	-0.26	73.3	-0.87	-	-	25	4.96

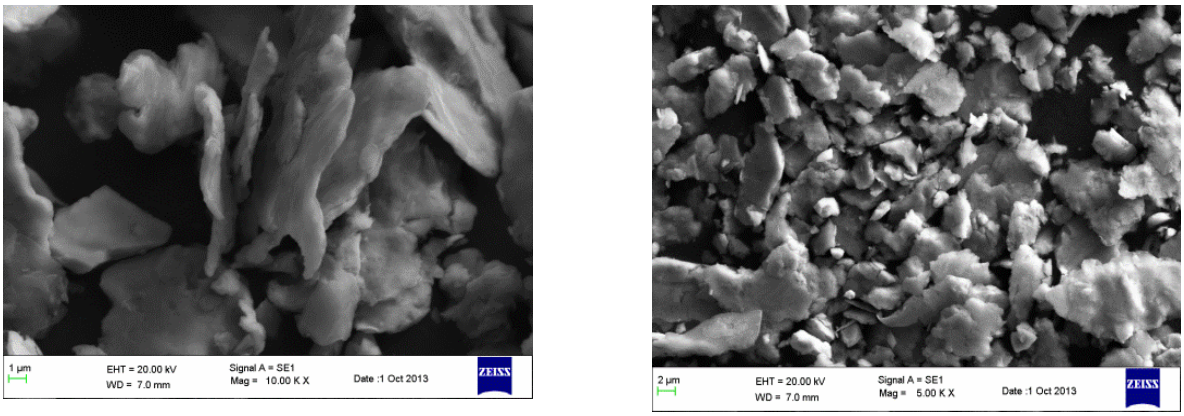


Fig. 2 SEM images of (a) CAM3 and (b) CAM18

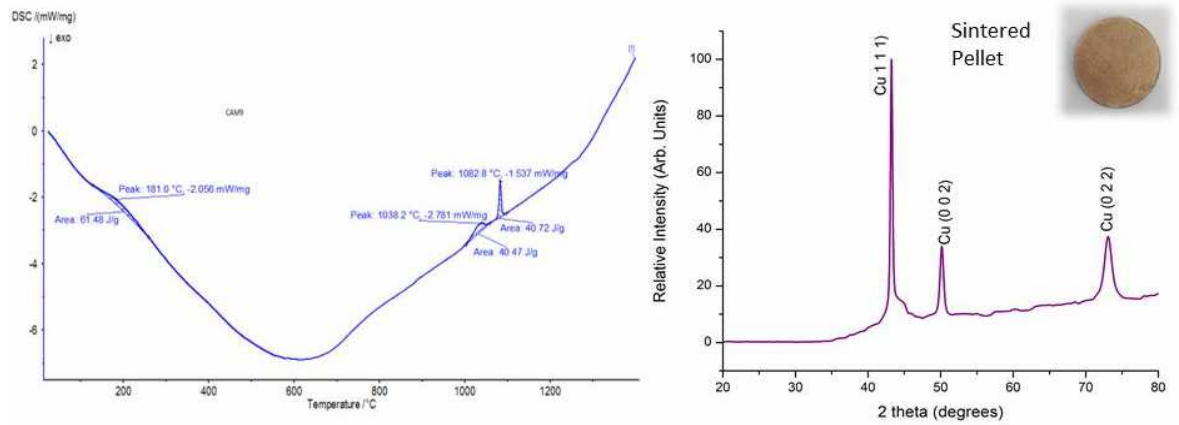


Fig. 3 (a) DSC curve of CAM9 powder; (b) XRD pattern and photograph of sintered pellet sample

Acknowledgements:

The authors would like to sincerely thank the Centre for Technological Development and Transfer (CTDT), Anna University for funding the project. The authors are grateful to Dr. S. S. Bhattacharya, Professor, MFL-MME, IIT Madras for permitting to use the lab facilities.

References:

1. Y. Sutou, T. Omori, J.J. Wang, R. Kainuma, K. Ishida, "Characteristics of Cu–Al–Mn-based shape memory alloys and their applications", Materials Science and Engineering A 378 (2004) 278–282.
2. M. Reza Rezvani, A. Shokuhfar, "Synthesis and characterization of nanostructured Cu–Al–Mn shape memory alloy by mechanical alloying", Materials Science and Engineering A 532 (2012) 282–286.
3. Richard A. Portier, Patrick Ochin, Alexandre Pasko, Gennady E. Monastyrsky, Andrei. V. Gilchuk, Victor I. Kolomytsev, Yuri. N. Koval, "Spark plasma sintering of Cu–Al–Ni shape memory alloy", Journal of Alloys and Compounds 266 (2012) 191–200.
4. R. Amini, S.M.M. Mousavizad, H. Abdollahpour, M. Ghaffari, M. Alizadeh, Ali K. Okyay, "Structural and microstructural phase evolution during mechano-synthesis of nanocrystalline/amorphous CuAlMn alloy powders", Advanced Powder Technology (2013) 1048-1053.

*****