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Novel Quaternary CuAlZnMg High Temperature Shape Memory Alloy (HTSMA) Fabricated by Minor Batch of Zn and Mg Additions

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Abstract: Shape memory alloys (SMAs) constitute the second largest commercial smart material class after piezoelectric materials. Different SMA alloy systems or SMAs with miscellaneous functionalities and characteristic properties have been designed for using in different applications until today. High temperature shape memory alloys (HTSMAs) are also widely desired to be used in various smart materials applications. HTSMAs with different functional and characteristic properties are muchly demanded for different tasks to be done by these alloys or devices designed by these alloys. A common and practical way to fabricate SMAs or HTSMAs with different shape memory effect (SME) and other properties is to fabricate them with different alloying compositions and add different additive elements. In this work, a quaternary CuAlZnMg HTSMA with an unprecedented composition consisting minor amount of zinc and magnesium additives was produced by arc melting method. As a result of applying post-homogenization in high β–phase temperature region and immediate quenching, the microstructural mechanism of a SME property was formed in the produced alloy. After then, to examine SME characteristics of the CuAlZnMg alloy some differential thermal analysis (DTA), microstructural (XRD) and magnetization (VSM) characterization tests were carried out. The DTA results showed that the alloy is a HTSMA exhibiting reverse martensitic transformations at temperature range between 167 °C and 489 °C. The XRD pattern obtained at room temperature revealed the martensite phases formed in the alloy, which phases are the base mechanism of the reversible martensitic transformation (the SME property) of the alloy. The VSM test showed that the alloy exhibit a diamagnetic property with a weak ferromagnetic coercivity contribution.

Key words: CuAlZnMg, High temperature shape memory alloy (HTSMA), Shape memory effect, Martensitic transformation, Enthalpy, DTA, XRD, VSM.

Minör Miktarda Zn ve Mg Katkıları Eklenerek Üretilmiş Yeni Kuaterner CuAlZnMg Yüksek Sıcaklık Şekil Hafızalı Alaşımı (YSŞHA)

Öz: Şekil hafızalı alaşımlar (ŞHA), piezoelektrik malzemelerden sonra ikinci en büyük ticari akıllı malzeme sınıfını oluşturmaktadır. Farklı SMA alaşım sistemleri ya da farklı işlevlere ve karakteristik özelliklere sahip SMA'lar bugüne kadar farklı uygulamalarda kullanılmak üzere tasarlanmıştır.Yüksek sıcaklık şekil hafızalı alaşımlar (YSŞHA) da farklı fonksiyonel ve karakteristik özelliklere sahip akıllı malzeme uygulamalarında yaygın olarak kullanılmaktadır. Bu alaşımlar veya bu alaşımlar ile tasarlanan cihazların farklı işlevleri için bu alaşımların farklı fonksiyon ve karakteristik özelliklere sahip olmaları çok talep edilmektedir. Farklı şekil hafıza etkisine (ŞHE) ve diğer özelliklere sahip ŞHA ya da YSŞHA'ları üretmenin yaygın ve kolay bir yolu farklı alaşım kompozisyonları ile ve farklı katkı elementleri ilave ederek üretmektir. Bu çalışmada, ark eritme yöntemi ile az miktarda çinko ve magnezyum katkı maddelerinden oluşan benzersiz bir kompozisyona sahip dörtlü CuAlZnMg YSŞHA üretilmiştir. Yüksek β-faz sıcaklık bölgesinde homojenleştirme ve hemen sonrasında hızlı söndürme işlemi uygulanması sonucunda alaşımda ŞHE özelliğinin mikroyapısal mekanizması oluşturulmuştur. Daha sonra, CuAlZnMg alaşımının ŞHE özelliklerini incelemek için diferansiyel termal analiz (DTA), mikroyapısal (XRD) ve magnetik karakterizasyon (VSM) gibi bazı karakterizasyon testleri yapılmıştır. DTA sonuçları, alaşımın 167 °C ve 489 °C sıcaklık aralığında ters martensitik dönüşümler gösteren YSŞHA olduğunu göstermiştir. Oda sıcaklığında elde edilen XRD desenleri, alaşımın tersinir martensitik dönüşümlerinin (ŞHE etkisi özelliğinin) temel mekanizmasını oluşturan alaşımdaki oluşmuş martensitik fazları ortaya çıkarmıştır. VSM testi alaşımın diyamanyetik özellik ve yanısıra buna ek olarak zayıf bir ferromanyetik koersivite sergilediğini göstermiştir.

Anahtar kelimeler: CuAlZnMg,Yüksek sıcaklık şekil hafızalı alaşım (YSŞHA), Şekil hafıza etkisi, Martensitik dönüşüm, Entalpi, DTA, XRD, VSM.

1. Introduction

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In terms of technological developments in the world investment and development of new materials is of great importance. These developments should not only be aimed and obtaining materials, and also these demands can be to reduce the cost of these versatile smart alloys or to increase their performance and change their properties [1–3]. Shape memory alloys (SMAs) are crystallographically designed smart materials that can make large distorsions like martensitic phase transformation in a temperature interval region or high elastic behavior at a higher temperature region which can be triggered by application of external effects such as heat or mechanical force, and can backtrack to their original shape when external forces are removed [4,5]. These exceptional behaviors of SMAs based on martensitic phase transformation are defined as their unique properties of shape memory effect (SME) and superelasticity (SE) [5–7]. These versatile alloys have already been utilized for their SME, SE and some other properties in numerous applications including actuator, automotive, aerospace, medical, robotics, micro/nano electromechanical systems (M/NEMS) and photodetectors [6,8–19].

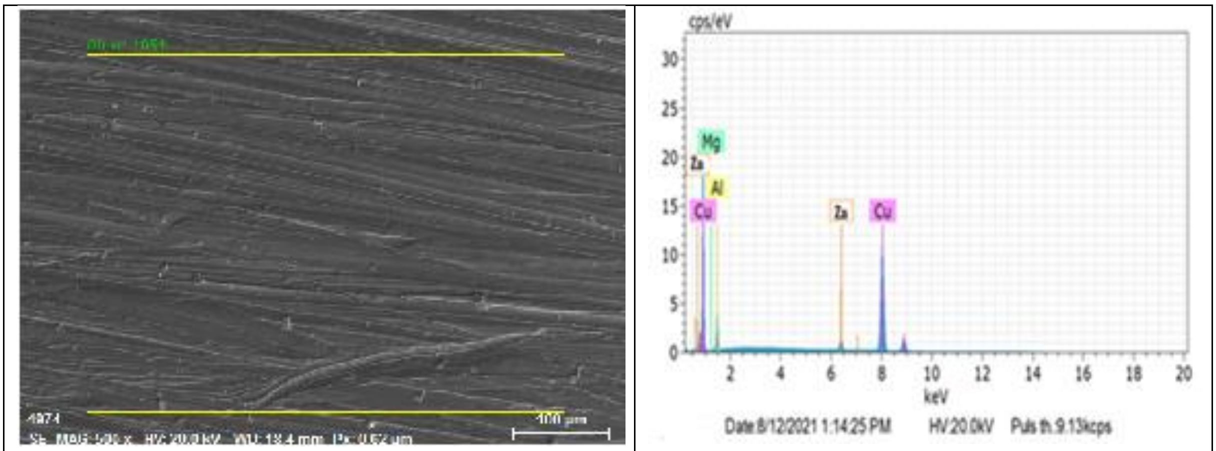
Martensitic phase transformations are solid-to-solid phase transitions that can occur isothermally and atomically a non-diffusional manner in SMAs between their two solid phases called as martensite (M) or product phase (at low temperature) and austenite (A) or parent phase (at high temperature) [5,8]. For a certain SMA that is commercial or ready to use in an application, one of the main characteristic parameters are its transformation or working (operation) temperatures which are the start and finish temperatures (M_f<M_s<A_s<A_f) and hysteresis gap (A_s-M_f) of phase transition reactions resulting martensite from austenite (direct transformation; A→M) upon cooling the alloy or austenite from martensite (reverse transformation; M→A) upon heating the alloy [5,8]. SMAs can show superealastic behavior at temperatures above their austenite finish temperature (A_f) untill a higher temperature (M_d) at which the martensite cannot be stress induced above this point [6,20].

SMAs are generally classified as; Cu-based, Fe-based and NiTi alloys [5,21]. Among these, NiTi alloys with superior shape memory properties are the most commercially used SMAs in industry and technological applications, but they are expensive and the production-processing processes are more difficult. This led researchers to pay attention on the second largest SMAs group; the Cu-based SMAs, which are regarded as the closest alternative to NiTi SMAs in terms of good SME and SE properties [7,22,23]. The most-known Cu-based SMAs such as Cu-Al-Ni, Cu-Zn-Al, Cu-Al-Mn alloy systems have been substantially studied untill today. The main reasons for the selection of Cu-based SMA systems are the low costs, easy fabrication and also much higher heat and electrical conducivity of these alloy systems as compared to NiTi ones. But, Cu-based SMAs have some drawbacks that are tried to be improved such as thermal instabilities, martensite stabilization and brittle nature and weak mechanical properties (low cold workability) stemmed from mainly their microstructural properties such as the large grain sizes, accumulation of secondary phases or impurities along the grain boundaries, high degree of order and also high elastic anisotropy in the β-phase [7,23–25]. A common and simple way to modify microstructure and reduce the grain size for improving these drawbacks and also to change characteristic martensitic transformation temperatures, SME, SE or other properties is to add some ternary, quaternary or more extra additive elements such as Ti, V, Co, Mn, Zr, Ce, Fe, Ni, B, Be, Mg, Sn or C [7,18,19,22–24,26–35]. SMAs are ultra sensitive to the compositional changes, their properties can change dramatically by even very little changes in the alloying composition. For example it was shown in a previous work [35] that the characteristic transformation temperatures of a CuAlMn SMA were decreased approximately 40-50 ºC by a quaternary 1.69 (at%) amount of magnesium addition which also caused formation of uniformly distributed spot-like Mg precipitations in the alloy due to the low solubility of Mg in Cu-matrix. In another work [36], the transformation temperatures of a CuAlMn SMA were reduced as nearly minimum 15 ºC and maximum 30 ºC by the addition of a 0.5 (wt%) magnesium content, and the strain recovery and superelasticity abilities were changed, too.

Although there are many studies been done on ternary CuZnAl SMAs [5,23,37–39] and some also on CuAlZn SMAs [27,40], there is no any Mg incorporated quaternary CuAlZnMg SMA work in the available literature. In this work, a CuAlZnMg SMA with a minor batch of Zn and Mg additives was produced by arc melting method. The characteristic microstructural properties, martensitic transformation temperatures and termodynamical parameters related to the shape memory properties of the alloy were investigated by differential calorimetry tests and structural measurements, and apart from these the magnetic properties of the alloy were tested by making vibrating sample magnetometer (VSM) measurement, too.

2. Experimental Details

In this study, the arc melting method was used to produce the quaternary CuAlZnMg alloy with an unprecedented composition of 74.79Cu-21.38Al-3.50Zn-0.34Mg (at%). At the beginning, the high purity (99.9%) of Cu, Al, Zn and Mg alloying elements powders were mixed, then the obtained mixture was pelletized under pressure. Then these pellets were melted together in a arc melter under argon atmosphere and the as-cast ingot alloy was obtained. Then the ingot was cut into small sized test samples (~4x4x2 mm and ~30-60 mg) proper for characterization measurements.Then in order to improve homogenization, these alloy samples were all heat-treated at 900 ˚C for 1 h in a furnace. At the end of this homogenization process, without waiting a pronto rapid cooling was made by quenching the alloy samples in iced-brine water solution. This traditional quenching method for this quick cooling of the alloy samples from β(A2) phase (to around 0 ºC) surpasses the formation of α and γ2 precipitation phases under eutectoid point and thus directly lead the formation of martensite phases in the alloy that will be the crystallographic base mechanism of shape memory effect property of the alloy. The chemical composition of the alloy was determined in room conditions by using a Zeiss Evo MA10 model EDS (energy dispersive X-ray spectrum). The SEM image and EDS result of the alloy are given in Fig.1.a and b, respectively. The diffraction planes of the martensite phases formed in the fabricated CuAlZnMg alloy were determined by performing structural X-ray diffraction (XRD) test using a Rigaku RadB-DMAX II diffractomer with CuKα radiation at room temperature. The DTA measurements were performed as taking three consecutive times of DTA heating/cooling cycles via using a Shimadzu DTG-60AH instrument between room temperature and 900 ˚C at a same single heating/cooling rate of 25 ˚C/min to observe the phase transitions occurred in the alloy in this temperature interval. The magnetic properties of the alloy were investigated by performing vibrating sample magnetometer (VSM) measurement by using a Quantum Design Physical Properties Measurement System (PPMS) with VSM under a magnetic field of ± 3 T at room temperature.



a) b)

Fig.-1. a) The SEM image and **b)** EDS spectra of the CuAlZnMg alloy at room temperature.

3. Results and Discussions

The DTA curves of the first, second and third heating/cooling cycles of the produced CuAlZnMg alloy each taken at the same single 25 ˚C/min of heating/cooling rate are given together in Fig.2. The emerged downside endothermic peaks and their corresponding upside exothermic counterparts observed on this DTA curves indicated the common multi-stage phase transitions behavior of Cu-based SMAs. Such that, this thermo-responsive events, which are all multi-stepped solid-to-solid phase transition reactions occurred either on heating and cooling the CuAlZnMg alloy, are caused by the heat induced structural and geometric changes in the crystal lattice of the alloy. As seen, from the far left of the downside heating segments of these DTA curves to their far right ends such multi-stage phase transitions arrays occurred as; martensite β1'(with γ1')→ austenite β1($L2_1$) → metastable β2(B2) → eutectoid dissolution of hypoeutectoid precipitations (α and γ2) → ordered β_2(B2) → disordered β(A2) on the each heating segments of the curves representing heating the CuAlZnMg alloy, and this phase transition array proceeded reversely way-back on cooling the alloy (as seen on the up-side cooling segments of the DTA curves), too [22,31,41–43]. To determine the characteristic reverse (M→A) and direct (A→M) martensitic transformation temperatures and some other transformational thermodynamic parameters of the CuAlZnMg alloy, the M→A and A→M phase transition peaks observed on the third cycling DTA curve were analyzed by DTA peak analysis program which applies tangent differentiation method automatically on a choosen peak area bordered manually and directly give as a data set of values of those transformational parameters. The determined parameter values of transformation temperatures and the values of some other related thermodynamic parameters hysteresis gap (A_s-M_f), A_{max} temperature, equilibrium temperature (T_0), enthalpy change ($\Delta H_{M\to A}$) and entropy change ($\Delta S_{M\to A}$) are given in Table 1. Here, the the equilibrium temperature, T_0 (calculated by using $T_0=(A_f+M_s)/2$ formula [7]) is the temperature at where the Gibbs free energy differences of two interconvertible solid (M and A) phases are equal therefore there is no any driving force to lead a martensitic transformation. Then, the entropy change amount, $\Delta S_{M\rightarrow A}$ was found by using $\Delta S_{M\rightarrow A}=\Delta H_{M\rightarrow A}/T_0$ formula [7,44]. As seen, the produced CuAlZnMg alloy is a high temperature shape memory alloy (HTSMA) [1,20,31,45] due to that it showed a reversible martensitic transformation at a temperature range (or simply an A_f temperature) far above 100 ºC.

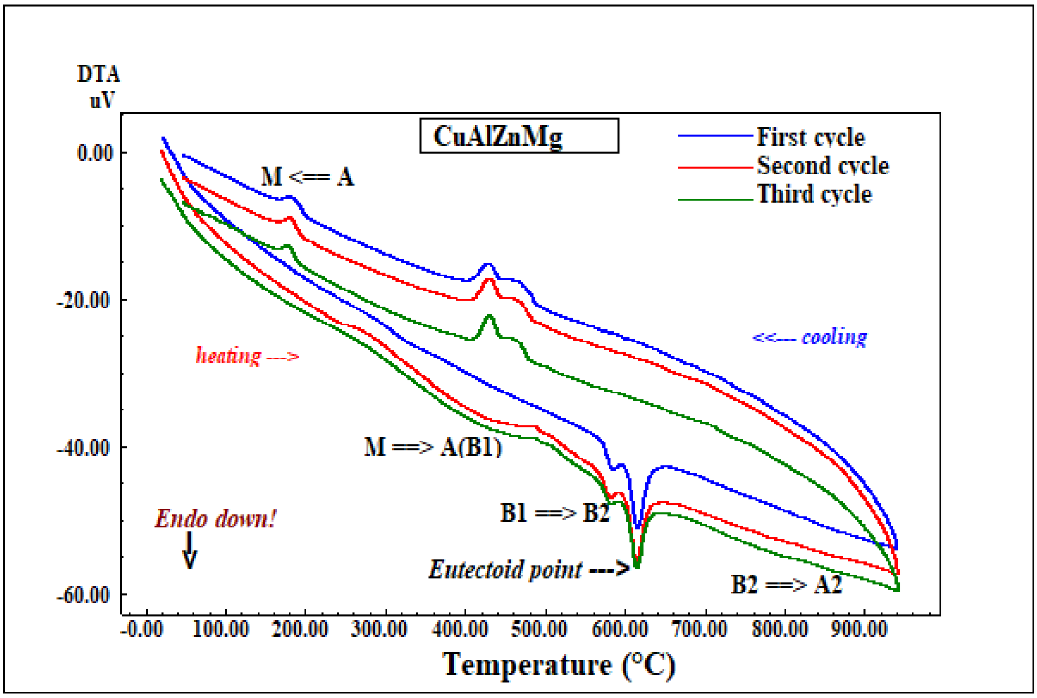


Fig.-2: The sequential cycling DTA curves of the CuAlZnMg HTSMA each taken at the same 25 °C/min of heating/cooling rate.

Table-1: The characteristic martensitic transformation temperatures and other transformational thermodynamic parameters values of the alloy determined by making the tangent peak analyses on the third cycle DTA curve.

A_s (ºC)	A_f (ºC)	A_{max} (ºC)	M_s (ºC)	M_f (ºC)	A_s -M_f (ºC)	T_0 (ºC)	$\Delta H_{M\rightarrow A}$ (j/g)	$\Delta S_{M\rightarrow A}$ (j/g)
316.69	489.30	382.11	190.31	167.08	149.61	339.81	14.16	0.042

Due to the some martensite stabilization, the endothermic peaks of the reverse transformation from martensite to austenite (M→A) phase observed on the down curve segments of heating the alloy were seen as abnormally too extended (i.e. Af-As=172.6 ºC) and therefore as seen in Table 1 the A_f temperature was found to be substantially elevated by the effect of minor Mg content. Some local stabilized martensite regions, which were formed by the heat effect of the first and second DTA cycles and residually remained, were needed more energies to be forced to transform to austenite and this extended this transformation till being fully completed. On the other hand the exothermic direct (A→M) transformation peaks observed on cooling the alloy are seen very stable. The enthalpy change ($\Delta H_{M\rightarrow A}$) value of the reverse transformation seen in Table 1 is very huge (14.16 j/gr) as compared to those (generally changing between ~1-12 j/gr) of other Cu-based SMAs [22,26,38,44,46,47], which indicates a martensitic transformation occurring by proceeding from difficult heat transmission pathways [22,42,44] and also a powerful shape memory effect property the produced CuAlZnMg alloy has.

The average valence or conduction electron number per atom (e/a) ratio of the Cu-based SMAs generally takes values between 1.45 and 1.51 to exhibit good shape memory effect [5,22]. The e/a ratio of the CuAlZnMg HTSMA was calculated as 1.466 and this value, been found indeed in the interval of 1.45- 1.51, theoretically indicates that a nearly high volume dominancy of β1’ (18R) over the co-existent γ1’(2H) type martensite phase with lower volume [42,48]. This theoretical expectation was confirmed by the microstructural XRD test result of the CuAlZnMg alloy given below.

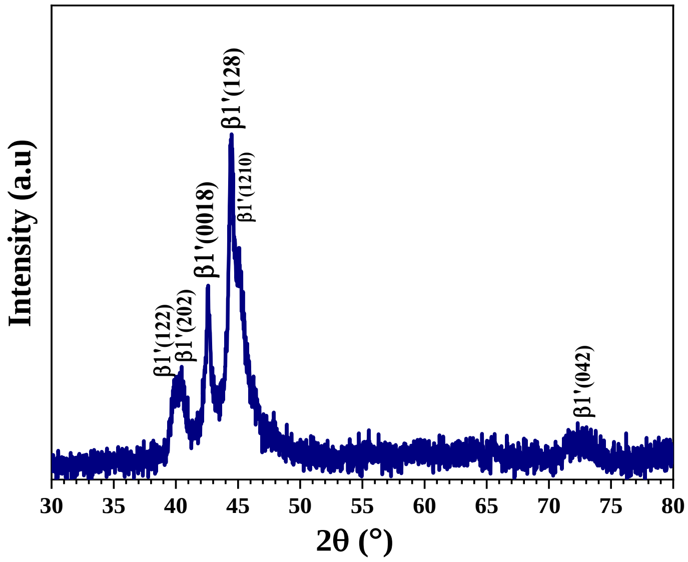


Fig.-3: The XRD pattern of the CuAlZnMg HTSMA obtained at room temperature.

The XRD pattern of the CuAlZnMg HTSMA obtained at room temperature is given in Fig.3. The main or the highest intensity peak is β1’(128) martensite peak and the others are β1’ martensite peaks of (122), (202), (0018),(1210) and (042) according to the reference works of [37,38,42,49]. The alloy has a polycrystalline structure. The crystallographic base mechanism of β1’ martensitic structure for the produced CuAlZnMg alloy to show a shape memory effect was found to be well formed in the alloy and this confirms the theoretical prediction made over the e/a ratio of this alloy mentioned above.

The magnetic hysteresis loop of the CuAlZnMg alloy in the form of magnetization (Ms) versus magnetic field strength (H) graph obtained by vibrating sample magnometry (VSM) test at room temperature (300 ˚K) is given in Fig.4. Here, magnetization (Ms) is magnetic moment per unit mass (emu/g). The VSM test showed a diamagnetic characteristic which stemmed from the main copper and minor zinc contents in the CuAlZnMg alloy [50–52]. A small inset graphic also inserted in Fig.4 shows the magnetic forcing hysteresis profile (coercivity) of the alloy between ±200 Oe magnetic field strength values and this means that the alloy has a very weak ferromagnetic property in addition to its diamagnetic property [7,50–54].

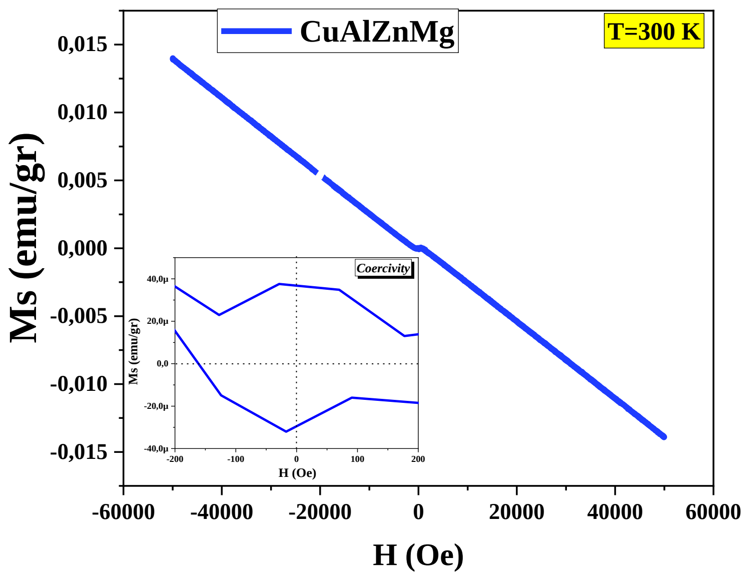


Fig.-4: Vibrating sample magnetometer (VSM) curve of CuAlZnMg HTSMA at room temperature.

4. Conclusions:

In this study, the novel CuAlZnMg high temperature shape memory alloy was succesfully produced by arc melting method. The DTA results showed that the alloy is a HTSMA exhibiting reversible martensitic transformations at a temperature range between 167 °C and 489 °C. The partial martensite stabilization remained from the prior DTA heating-cooling processes led the reverse martensitic transformation to be extended and thus the A_f temperature of the alloy was found to be increased. The average conduction electron ratio (e/a) of the alloy was found as 1.466 which indicates the martensite phases should have formed in the alloy to show a shape memory behavior. The existence of martensite phases was revealed by XRD pattern of the alloy obtained at room temperature, which phases are the base mechanism of the reversible martensitic transformation (the SME property) of the alloy.The VSM test showed that the alloy has a diamagnetic nature with a weak ferromagnetic coercivity contribution. In conclusion, the novel CuAlZnMg high temperature shape memory alloy with good shape memory effect property was produced and may be useful in related HTSMA applications and further research on HTSMAs.

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