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Thermal expansion, electrical conductivity and hardness of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites

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Abstract: $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites were successfully prepared by sintering at 623 K and 723 K. It is found that the thermal expansion of Al is effectively decreased with the addition of negative thermal expansion material $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$, which plays the role of the thermal expansion compensator in the composite system. The thermal expansion of composites can be tailored by adjusting the volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$. In particular, 40 vol.-% $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite sintered at 723 K has a low thermal expansion coefficient of $2.38 \times 10^{-6} \text{ K}^{-1}$. The thermal expansion of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites matches well with those calculated from the rule of mixture (ROM) estimation. The ROM model can be used to predict the thermal expansion of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite and design $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite with a particular thermal expansion. $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites exhibit excellent electrical conductivities at the level of 10^4 S/cm . The hardness of the as-prepared composites increases by adding $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ in Al.

Keywords: electrical properties; metal-matrix composites; sintering; thermal properties.

1 Introduction

Most materials have a positive thermal expansion (PTE) coefficient, expanding on heating and contracting on

cooling. However, some materials have abnormal thermal expansion properties, which contract on heating, expand on cooling, and have negative thermal expansion (NTE) coefficient. A new NTE material, Mn_3MN ($M=\text{Cu}$, Zn , Ge and Sn), has been discovered in past decades [1–5]. The antiperovskite manganese nitrides display an isotropic NTE at least twice than that of the well-known NTE material ZrW_2O_8 (the coefficient of thermal expansion, $\alpha = -8.9 \times 10^{-6} \text{ K}^{-1}$) [2, 6, 7]. Besides, these NTE materials have good mechanical and physical properties, such as Young's modulus, hardness, thermal conductivity, electrical conductivity and magnetic properties [8–11]. The composites with low or zero thermal expansion by blending PTE materials with NTE materials, will improve the mechanical reliability and increase the long-term duration of devices and instruments applied in high precision industries, such as optics, energy transformation and microelectronics.

In recent years, the composites with controllable thermal expansion have been fabricated by mixing ZrW_2O_8 with some PTE materials, such as Al [12], Cu [13], SnO_2 [14], ZrO_2 [15], $\text{Zr}_2\text{WP}_2\text{O}_{12}$ [16], cement [17] and polyimide [18]. Aluminum has an easy process ability, low density, excellent thermal and electrical conductivity, but the large PTE ($22.2 \times 10^{-6} \text{ K}^{-1}$) restricts its application in the field of microelectronics and precision instruments. Matsumoto et al. [12] successfully fabricated $\text{ZrW}_2\text{O}_8/\text{Al}$ composite with a thermal expansion near to zero, but the volume fraction of ZrW_2O_8 was up to 75%, consequently, the mechanical and physical properties of the composite are worsen. Furthermore, it was reported that the decomposition of ZrW_2O_8 appeared in Al matrix at the temperature of about 683 K, which degraded the performance of thermal expansion compensation [19]. $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$, as a member of the antiperovskite manganese nitrides, displays a large isotropic NTE and the magnitude of its net NTE is close to the PTE of metals, which can be used to prepare a low thermal expansion composite with a low volume fraction of manganese nitride in metal matrix. To the best of our knowledge, the composites comprising of the antiperovskite manganese nitrides were rarely reported [9, 20, 21]. Therefore, it is necessary and important to study the thermal expansion and

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Table 1: Physical properties of metal matrix and thermal expansion compensator.

Material	Coefficient of thermal expansion α ($\times 10^{-6} \text{ K}^{-1}$)	Young's modulus E (GPa)	Electrical conductivity σ (S/cm)	Vickers hardness HV (kgf/mm ²)
Al	23.2	70	3.77×10^5	29
$\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$	-19.16	200 [8]	0.157×10^3	538

other physical properties of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites for the potential practical application in microelectronics and precision instruments.

In this work, the composites containing $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ and Al were prepared using a solid-state sintering method. The sintering temperature (T_s) of composites was determined. The thermal expansion of Al was effectively decreased by the thermal expansion compensator (TEC) $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ and the thermal expansion of composites was tailored by adjusting the volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$. The electrical conductivity and hardness of TEC and composites were also investigated at room temperature. The physical properties of the metal matrix and the TEC are listed in Table 1.

2 Experimental procedures

The polycrystalline $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ was synthesized by a solid state reaction using the powders of the synthesized Mn_2N [9], Zn (99.99 at.%) and Sn (99.99 at.%) as raw materials. The powders were mixed with the desired proportions and milled by a planetary ball mill machine (P5, Fritsch, Germany) at a rotating speed of 250 rpm for 2 h. Then the milled powders were pressed into cylindrical pellets. The pellets were fired at 1073 K under the flowing argon for 20 h to prepare $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$. The obtained $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ was pestled in agate mortar for 1 h and further ground by mechanical ball milling at a rotating speed of 300 rpm for 20 h. The milled nitrides powders were mixed with the commercial Al powders in appropriate volume ratios by mechanical ball milling at a rotating speed of 250 rpm for 20 h. The mixed powders were pressed into a cylindrical shape by cold isostatic pressing process. The cylindrical pellets were sintered at 623 K, 723 K and 823 K under the flowing argon shield for 3 h, respectively, and then cooled down to room temperature.

The crystal structures of the samples were investigated by the X-ray powder diffraction (Rigaku D-max2500/pc, Japan) using $\text{Cu } K_\alpha$ radiation, at a scanning rate of $5^\circ/\text{min}$ from 20° to 80° . The linear thermal expansion of samples was measured by thermal

mechanical analyzer (Netzsch TMA 402F3, Germany) in the temperature range of 290–400 K. The electrical conductivity was measured by a standard four-probe technique (RTS-9, China) at room temperature. The Vickers hardness was obtained by Vickers hardness tester (HV-1000, China).

3 Results and discussion

3.1 Analysis of the $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$

The X-ray diffraction (XRD) pattern of the as-prepared $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ is shown in Figure 1, which indicates a typical antiperovskite cubic structure (space group $Pm\bar{3}m$, JCPDS card No.23-0220). The existence of MnO is likely due to the oxygen contamination introduced by the ball milling, which has little influence on the thermal expansion of the antiperovskite manganese nitrides [9]. Figure 2 displays linear thermal expansion $\Delta L/L$ (298 K) of the TEC $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$. It reveals that $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ displays a NTE at 301.3–363.2 K ($\Delta T = 62 \text{ K}$) and the coefficient of linear thermal expansion α obtained by linear fitting is $-19.16 \times 10^{-6} \text{ K}^{-1}$, which is more than twice as that of ZrW_2O_8 [7]. The electrical conductivity and Vickers hardness is 157 S/cm and 538 HV (Table 1), respectively.

Figure 1: X-ray diffraction pattern of the thermal expansion compensator $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$.

Figure 2: Linear thermal expansion of the thermal expansion compensator $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$.

3.2 Analysis of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite

The sintering temperature T_s is a key factor in fabricating $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites. It was reported that the sintering temperature of Al with high-density is above 573 K but lower than its melting point T_m (933 K)

Figure 3: X-ray diffraction patterns of 30 vol.-%- $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites at (A) 623 K, (B) 723 K, (C) 823 K. The patterns of matrix Al (D) and compensator $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ (E) are added as well for comparison.

[20]. The carbon nanotubes/manganese nitrides composites have been successfully fabricated at 1223 K in our previous study [9]. Here, the prefabricated columnar samples of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites were sintered at 623 K, 723 K and 823 K, respectively, obtaining the dense composite samples with the powders well-distributed in the matrix Al. Figure 3 displays the XRD patterns of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$, aluminum and the as-prepared 30 vol.-%- $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites after sintering at 623 K, 723 K and 823 K, respectively. When T_s are 623 K and 723 K, all of the diffraction peaks could be indexed as the characteristic peaks of either $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ or Al, indicating that no chemical reaction between the Al matrix and nitride compensator occurred during the sintering. However, when T_s is 823 K, the XRD pattern is different from those of the above two, and the characteristic peaks of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ disappear, accompanying with appearance of unknown phase peaks. The characteristic peaks of the unknown phase do not match that of the reported Mn_3AlN [22] or other antiperovskite manganese compounds.

The thermal expansion of the obtained specimens was measured at the temperature range of 290–400 K. Figure 4 shows linear thermal expansion of 30 vol.-%- $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites specimens sintered at 623 K, 723 K and 823 K, respectively. For the samples sintered at 623 K and 723 K, an abnormal change of thermal expansion is observed at the NTE temperature range of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$, though the specimens still display a PTE. The thermal expansion of composites is decreased by mixing the TEC in the Al matrix, resulting in a low thermal expansion about $5.5 \times 10^{-6} \text{ K}^{-1}$ ($T_s = 723 \text{ K}$) at 301.3–363.2 K. It was reported that the NTE decreased with increasing the sintering temperature for the antiperovskite manganese nitrides [23]. In this study, both composites sintered at

Figure 4: Linear thermal expansion of 30 vol.-%- $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites at different sintering temperatures.

623 K and 723 K show almost similar thermal expansion at the measured temperature range. It indicates that the relatively low sintering temperature for $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite has little influence on thermal expansion, which is conducive to fabricating fine low thermal expansion $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite. However, for the sample sintered at 823 K, the $\Delta L/L$ of the 30 vol.-% $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ specimen is kept as a linear dependence with temperature over the measured temperature range, due to the unknown phases existed in composite.

Based on the results above, the $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites were fabricated at 723 K. The 30 vol.-% $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite displays a low thermal expansion compared with that of the Al matrix. It can be expected to obtain the $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite with particular thermal expansion by adjusting the volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$. When the volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ is up to 40%, it can be observed that 40 vol.-% $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite displays a low thermal expansion ($\alpha = 2.38 \times 10^{-6} \text{ K}^{-1}$, at $T = 301.3\text{--}363.2 \text{ K}$) in Figure 5.

The thermal expansion of composite can be evaluated using two models, such as the rule of mixture (ROM) and *Turner's* model [24]. Here, we define the subscripts c, m and t as the composite, matrix and TEC, respectively. Therefore, the coefficient of thermal expansion of composite, matrix and TEC can be described as α_c , α_m and α_t , respectively. Thermal stress is assumed uniform throughout the composite for ROM, which means that the thermal expansion of matrix and compensator is independent. The thermal expansion of composite is represented by the volume-weighted sum of contributions from the matrix and the dispersed phase, which is described as Equation (1):

$$\alpha_c = v_m \alpha_m + v_t \alpha_t, \quad (1)$$

Figure 5: Linear thermal expansion of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites for various $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ volume fractions.

where v_m and v_t donates as the volume fractions of matrix and compensator, respectively, and $v_m + v_t = 1$. For *Turner's* model, the thermal strain is assumed approximately uniform throughout the composite as a result of interfacial elastic interactions between the matrix and compensator, which is described as Equation (2):

$$\alpha_c = \frac{v_m E_m \alpha_m + v_t E_t \alpha_t}{v_m E_m + v_t E_t}, \quad (2)$$

where E_m and E_t is Young's modulus of matrix and compensator, respectively. It is confirmed that the larger elastic modulus of constituent contributes more to thermal expansion than the volume fraction in *Turner's* model. Figure 6 displays the experimental value of $\Delta L/L$ as well as the curves calculated by assuming ROM and *Turner's* model. It can be observed that the experimental value of $\Delta L/L$ almost coincides with the value calculated from ROM for the obtained 30 vol.-% $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite. Therefore, the volume fraction, instead of the high stiffness of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ in this composite, dominates the contribution to suppressing thermal expansion. The ROM can be used to predict the thermal expansion of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite and design $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite with a particular thermal expansion.

As listed in Table 2, the electrical conductivity and hardness of the composite are measured. It can be seen that the average electrical conductivities of the obtained composites are all at the level of 10^4 S/cm , because $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ is an electrical nonconductor compared with Al. The electrical conductivity decreased significantly with increasing the volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$. For example, the electrical conductivity of 40 vol.-% $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite is less than half of that of

Figure 6: Experimental plots of thermal expansion as well as curves calculated by assuming ROM and *Turner's* model for 30 vol.-% $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite.

Table 2: The average electrical conductivity and Vickers hardness of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites.

Volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$	Sintering temperature (K)	Electrical conductivity (10^4 S/cm)	Hardness (HV)
30%	623	8.02	56.2
	723	7.94	60.5
40%	623	3.63	78.3
	723	3.60	84.6

30 vol.-%- $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite even though the volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ increases 10%, which indicates that volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ has a significant effect on the electrical conductivity of the composite. It can be found that the electrical conductivity decreased slightly with increasing sintering temperature. The sintering temperature, far less than the volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$, has a slight effect on electrical conductivity.

It was reported that *Pal* developed a new theoretical model and optimized using a differential effective medium approach for accurately evaluating the electrical conductivity of composite [24], which is described as Equation (3):

$$\left(\frac{\sigma}{\sigma_m}\right)^{\frac{1}{3}} \left(\frac{\sigma_d - \sigma_m}{\sigma_d - \sigma}\right) = \left(1 - \frac{v}{v_m}\right)^{-\mu v_{\max}} \quad (3)$$

where σ , σ_m and σ_d donates as the electrical conductivity of composite, matrix and dispersed phase, respectively, and v_d , $v_{\max}$ and μ is the volume fraction, max volume fraction of dispersed phase and correction factor of the order of unity to account for the deviations from the assumptions, respectively. We define the relative electrical conductivity σ_r and the electrical conductivity ratio λ as σ/σ_m and σ_d/σ_m , respectively. It is known that λ (less than 0.01) approaches 0. Therefore, the Equation (3) can be simplified to Equation (4):

$$\sigma_r = \left(1 - \frac{v_d}{v_{\max}}\right)^{\frac{3}{2}\mu v_{\max}}. \quad (4)$$

Here, $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ particles are assumed to have the regular spherical shape, and $v_{\max}$ is 0.637. Figure 7 shows the experimental average electrical conductivity and the curve predicted by assuming of electrical conductivity for $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites. When μ is 2.5, the experimental data of electrical conductivity coincides with the *Pal*'s theoretical model. It can be found that the irregular

Figure 7: Experimental data of electrical conductivity and curve calculated by assuming *Pal*'s theoretical model for $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites.

spherical particles, distribution of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ particles, interfacial additive and other correction factors lead a relatively larger μ , and the preparation of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composite can be optimized in further research.

The Vickers hardness of Al and $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$ is 29 HV and 538 HV, respectively (load of 4.9 N, Table 1). The function of the compensator is not only decreasing the thermal expansion but also improving the hardness of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites. The hardness of composite with a certain volume fraction increases with increasing the sintering temperature. For example, the Vickers hardness of 30 vol.-%- $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ sintered at 723 K (60.5 HV) is about 10% than that of 30 vol.-%- $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ sintered at 623 K (56.2 HV), which could be caused by the increase of interface bonding with increasing temperature. When the composite is sintered at the certain temperature, the Vickers hardness of the composites increases efficiently with increasing the volume fraction of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$, which is owing to the effective suppression of plastic deformation of matrix from the increasing the volume fraction of compensator.

4 Conclusions

$\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites were successfully prepared by sintering at 623 K and 723 K. The sintering temperature has little influence on the thermal expansion of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites. The thermal expansion of composite decreases effectively by adding the compensator in the NTE temperature range of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$. The coefficient of thermal expansion of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites is tailored by adjusting the volume fraction of

$\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}$. The thermal expansion of $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites agree well with that calculated from the ROM estimation. $\text{Mn}_3\text{Zn}_{0.5}\text{Sn}_{0.5}\text{N}/\text{Al}$ composites exhibit excellent electrical conductivity and improved hardness for the potential application in the field of microelectronics and precision instruments.

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