

Thermal expansion behavior and mechanical properties of zirconia toughened alumina reinforced high chromium cast iron matrix composites

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Abstract: A method based on squeeze casting technology, without the requirement of pre-coating for the particles and the usage of preforms, was proposed for the preparation of particle-reinforced metal matrix composites. Zirconia toughened alumina (ZTA) particles reinforced high chromium cast iron (HCCI) matrix composites were fabricated using this method. The microstructure, thermal expansion behavior, and properties including hardness, Young's modulus, and strain recovery of the prepared composites were investigated by means of SEM, XRD, thermal mechanical analyzer, and nanoindentation tests. The results illustrate that the particles are relatively uniform distributed within the matrix and a mechanically strong bonding interface is obtained. The experimentally determined average coefficient of thermal expansion (CTE) of the composites is $12.77 \times 10^{-6} \text{ K}^{-1}$, which is in good agreement with theoretically calculated values, showing an error of less than 2%. The hardness and Young's modulus of the ZTA are higher than that of the interface and matrix, and the elastic and plastic behavior of the interface is close to that of the ZTA particles.

Keywords: iron matrix composites; ZTA ceramic; thermal expansion; nanoindentation

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1 Introduction

ZrO₂ toughened Al₂O₃ ceramic (ZTA) particles, known for their exceptional strength, toughness, and hardness, are widely employed in various industry fields^[1-3]. Particularly, an increasing number of research works have focused on introducing ZTA as a reinforcing phase into iron matrix to improve the wear-resistant properties of metal materials^[4-6]. At present, iron composites reinforced with ZTA particles are generally applied to substitute conventional metal materials in many engineering areas, including cement, construction, and mining industries, etc.

The techniques utilized for preparing these composites mainly comprise the infiltration casting method and powder metallurgy. For the former process, the composites are fabricated by infiltrating molten metal into the porous ceramic precursor. Researchers such as Sui et al.^[7] and Zheng et al.^[8] manufactured ZTA/iron composites using gravity casting infiltration and lost foam casting infiltration, leading to a notable enhancement in the three-body abrasive wear performance compared to the iron matrix. Li et al.^[9] introduced ZTA preforms into Cr15 matrix to realize the pressureless infiltration preparation of the composites, and the results illustrated that the relative erosive wear resistance of the composites was significantly higher (1.68–2.43 times) than that of the iron matrix. Additionally, the three-body abrasive wear resistance of the composites was determined to be 1.64–2.21 times greater than that of the iron matrix^[10]. Although the casting infiltration technology is widely used and some progresses are achieved, challenges including limited infiltration capacity,

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issues with material purity and the inherent casting defects still remain^[11]. In addition to the liquid infiltration method, powder metallurgy technology is also employed for the preparation of particles-reinforced composites. Chen^[12, 13] obtained ZTA/Fe45 composites through vacuum sintering process, where ZTA and Fe45 alloy powder were mixed, compacted and then sintered to create the composites. The results of impact abrasive wear behavior indicated that the composites exhibited superior wear resistance, achieving up to 1.88 times the maximum wear resistance compared to the matrix materials^[14]. However, this method is not suitable for manufacturing large parts, meanwhile, the required equipment is expensive and the size of the prepared product is limited, making it difficult to industrialize.

Considering the drawbacks exists in infiltration casting and powder metallurgy methods, they are not conducive for industrial applications. In current study, a technology based on squeeze casting was employed to prepare ZTA-reinforced iron composites. This method involves two critical steps, namely, flow mixing of liquid metal and ZTA and pressure bonding between ZTA and matrix, aimed at ensuring the uniform distribution of ceramic particles and establishing strong interfacial bonding. The detailed procedure and the impact wear performance of the prepared ZTA/iron composites have been mentioned in previous works^[15, 16].

Although the wear resistance and mechanical properties of iron matrix composites have been extensively researched, the thermal behavior of these composites, especially those fabricated using this method other than infiltration casting and powder metallurgy techniques, has received comparatively less attention. Actually, the application of metal matrix composites as structural materials depends on their adequate and comprehensive properties to withstand various mechanical and thermal conditions^[17]. Among the different thermal behavior characteristics, the thermal expansion behavior of composites stands out as one of the most widely studied attributes because it not only provides insights into thermal expansion, but also reveals information on potential thermal stresses that could affect the mechanical properties of the composite in complex thermal environments. Moreover, given the inherent thermal mismatch between the reinforcing particles and the iron

matrix, understanding how these materials respond to different thermal conditions that might encountered during service is crucial. Consequently, the long-term thermal behavior and stability of the composites emerge as critical considerations in the design process^[18].

In this work, a method based on squeeze casting was utilized to prepare ZTA/HCCI (high chromium cast iron) composites. The microstructure and thermal expansion behavior of the fabricated composites were investigated, and the experimental results of thermal expansion were compared with the predicted results through various theoretical models. In addition, the hardness, modulus, and strain recovery of the composites were studied through nanoindentation test.

2 Experimental procedures

The micro-alloyed HCCI, with chemical compositions of 24.53% Cr, 2.75% C, 1% Mn and Ni, 0.98% Si, 0.97% W, 0.75% Mo, and balance Fe (wt.%), was used as the matrix alloy. Commercially available ZTA particles (2–3 mm) with a composition of 60% Al₂O₃ and 40% ZrO₂ (vol.%) were adopted as the reinforcing phase, characterized by a thermal expansion coefficient of 7.5×10^{-6} – 9.5×10^{-6} °C, a hardness of 1,700 HV, and a fracture toughness of 7.0 MPa·m^{1/2}. The morphology [Figs. 1(a–b)] and XRD pattern [Fig. 1(c)] of the ZTA particles are illustrated in Fig. 1, where the particles exhibit irregular shapes.

The preparation process of the ZTA/HCCI composites is shown in Fig. 2. The homogeneous distribution of the ZTA particles within the matrix was achieved by rapid flowing liquid metal encapsulating the particles and mixing with them under filling pressure. Then, the interfacial bonding between the ZTA and matrix was established through applying high boost pressure to the ceramic and metal mixture during solidification process. The experimental details have been reported in previous studies^[15, 16]. Prior to preparation, the HCCI was heated to a temperature exceeding 1,500 °C for melting, while the ZTA particles were preheated to 1,000 °C to enhance their surface activity. Subsequently, the molten metal and preheated particles were individually poured into

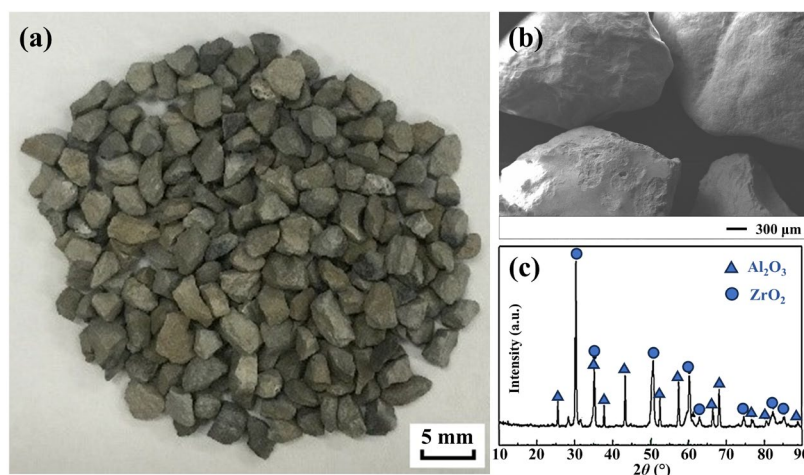


Fig. 1: Morphology (a–b) and XRD pattern (c) of ZTA particles

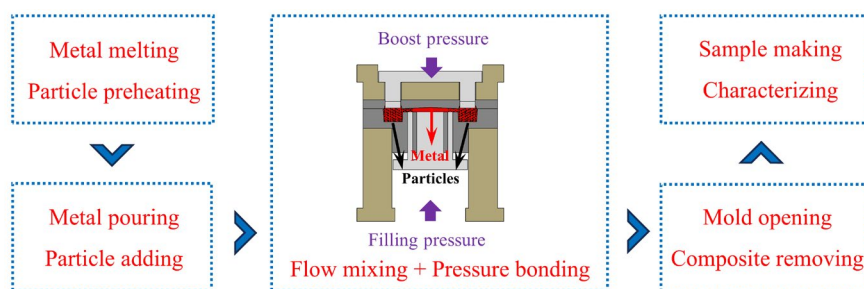


Fig. 2: Preparation process of ZTA/HCCI composites

the mold. After that, a filling pressure was applied, enabling the mixing of liquid metal and particles in the filling channel and then the mixture was pushed into the cavities, this process was referred to as flow mixing. Then, the mixture of metal and particles was subjected to further solidified and bonded closer under a boost pressure, known as pressure bonding. After the solidification was finished, the pressures are removed and the mold was opened to extract the composites parts.

The metallographic samples (8 mm×8 mm×10 mm), thermal expansion samples (25 mm×6 mm×6 mm), and nanoindentation samples (8 mm×8 mm×10 mm) were cut from the prepared materials using a metallographic cutting machine. Subsequently, the metallographic samples were polished following standard procedures and then etched with a 4% nital solution to reveal the microstructure. The microstructure of the samples was observed using a SEM (EVO 18) equipped with an EDS (energy dispersive spectrometer). The phase constitutions of the samples were analyzed via XRD (Ultima IV diffractometer with Cu K α radiation).

In addition, the coefficient of thermal expansion (CTE) of the composites was determined using a DIL 402CL thermal mechanical analyzer with a measurable temperature range of 20–1,500 °C and a measurement sensitivity of $dL/L_0 > 10^{-4}$. The test parameters were set as follows: temperature range: 25–1,050 °C, heating rate: 5 K·min⁻¹, cooling rate: 15 K·min⁻¹, sample holder material: Al₂O₃, protective atmosphere: Ar gas. Prior to the test, the equipment was calibrated with Al₂O₃ to eliminate systematic errors. Three samples experienced examination and the average value was adapted for analysis.

The measurement of CTE was based on the length change of the sample as the temperature increased, and the curve of the sample length as a function of temperature was recorded by the thermal expansion meter. The average coefficient of linear expansion across various temperature ranges can be determined using Eq. (1):

$$\alpha = \frac{\Delta L}{L_0 \Delta T} \quad (1)$$

where α represents the linear thermal expansion coefficient of the material (10⁻⁶ K⁻¹), L_0 denotes the initial length of the sample (mm), ΔT indicates the range of temperature change (°C), and ΔL signifies the length change of the sample over a temperature interval ΔT (mm).

Based on the “Oliver-Pharr” method^[19], the hardness (H) and Young’s modulus (E) of the composites were tested using a nanoindentation system (Agilent, G200) equipped with a

Berkovich tricone indenter. Meanwhile, the depth of loading displacement was set at 400 nm and the approach velocity was maintained at 10 nm·s⁻¹.

3 Results and discussion

3.1 Microstructure of composites

The distribution of the particles within the matrix and the interface status between the ZTA and matrix are depicted in Figs. 3(a) and (b), respectively. It can be seen that the ceramic particles are relatively uniform distributed inside the matrix, and the original appearance is maintained, where exhibits an irregular shape. Meanwhile, the statistical analysis through the Image-Pro Plus software reveals that the volume fraction of the particles is approximately 50%.

The microstructure of the ZTA and HCCI in the composites is shown in Figs. 3(a')–(a''), respectively. The former, namely, ZTA exhibits dendritic ZrO₂ dispersed within the Al₂O₃ substrate, which enhances its toughness and strength through the phase transition and lattice confinement effects of ZrO₂^[20]. The distribution of elements Al, Zr, and O through surface scanning is also given below Fig. 3(a'). Additionally, the microstructure of HCCI in Fig. 3(a'') illustrates a typical dendritic structure, with strip-shaped (Fe, Cr)₇C₃ carbides distributed in the austenitic substrate (γ -Fe). A small quantity of martensite α -Fe (resulting from γ -Fe transformation during cooling process) is also found at the boundary of γ -Fe and carbides. Moreover, the fine grain size of (Fe, Cr)₇C₃ (<10 μ m) and γ -Fe (<50 μ m) can be attributed to the exertion of a higher pressure. The elemental distribution of Fe, Cr, and C in the metal matrix is also provided.

The interface exhibits continuous, tight, and distinct characteristics [Fig. 3(b)] without defects such as delamination or detachment. According to the results of the EDS line scanning across the interface in Fig. 3(b'), the distribution curves of different elements include Fe, Cr in metal area and Al, Zr, O in ceramic region are drastically changed at the margin of interface. Meanwhile, there is no evidence of elements in metal diffusing into the ceramic side or elements in ceramic spreading into metal area. Moreover, no interface products were detected, further demonstrating that the bonding mode of the interface is a mechanical bonding state.

In order to identify the phases present in the prepared composites, XRD analysis was conducted and the result is presented in Fig. 3(c). XRD result illustrates that the main

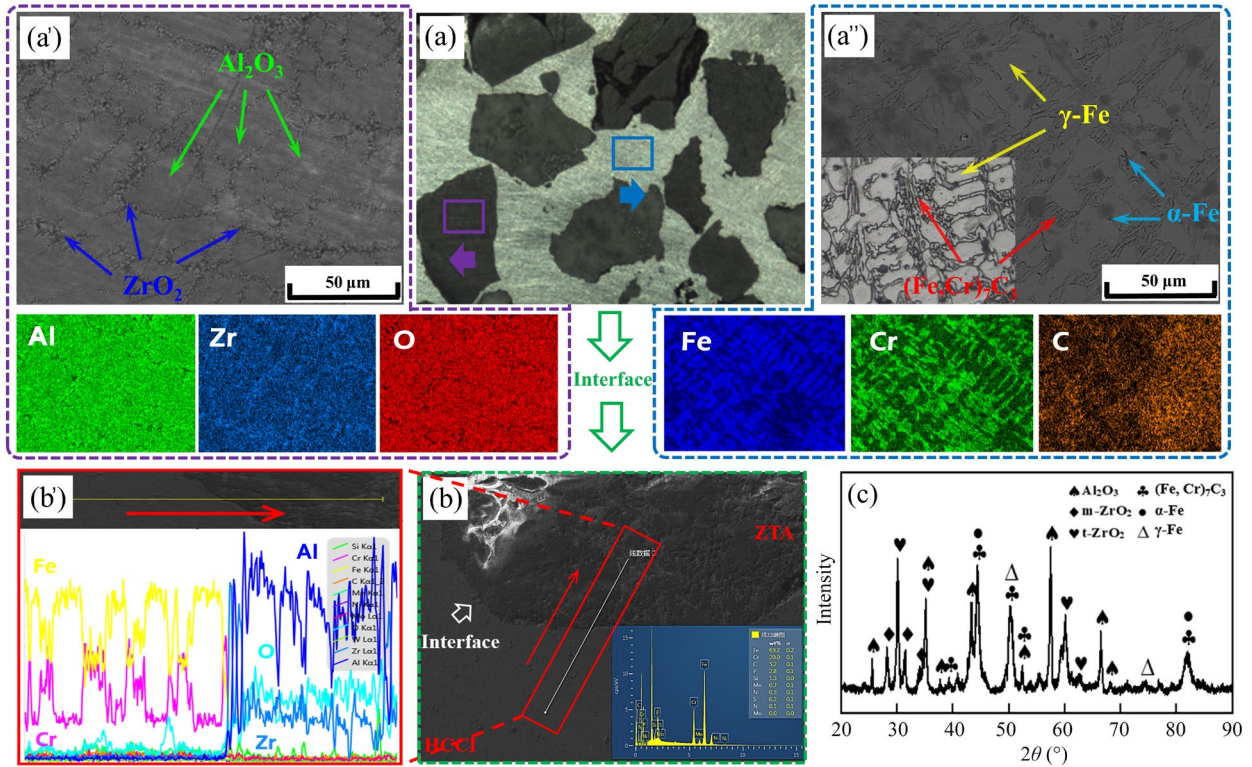
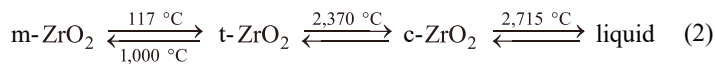


Fig. 3: Particle distribution within matrix (a), microstructures of ZTA and HCCI (a')-(a''), interfacial bonding characterization (b)-(b'), and XRD analysis results of composites (c)

phases of the composites consist of Al₂O₃, m-ZrO₂, t-ZrO₂, (Fe, Cr)₇C₃, γ-Fe, and α-Fe. The martensite α-Fe is formed through the transformation of austenite γ-Fe during cooling and solidification. Owing to the presence of carbide-forming elements, part of the carbon is absorbed to create carbides, resulting in a carbon-deficient region surrounding the austenite. Additionally, the decrease in carbon and chromium solubility caused by the diffusion of some carbon and chromium into the eutectic carbides also contributes to carbon and chromium depletion around the austenitic. While the reduced carbon content can elevate the transformation temperature of martensite, causing austenite to partially transform into martensite during cooling and solidification^[21].

In ZTA ceramics, the ZrO₂ phase experiences three crystalline phase transitions during the heating and cooling processes: the monoclinic phase (m-ZrO₂) at lower temperatures, the tetragonal phase (t-ZrO₂) at medium temperatures, and the cubic phase (c-ZrO₂) at higher temperatures. The transition relationships of the three crystal types are outlined as follows^[22]:



In this study, the melting temperature of metal exceeds 1,500 °C, and the ZrO₂ experiences a reversible phase transition from the monoclinic phase (m-ZrO₂) to the tetragonal phase (t-ZrO₂), which accompanies by a certain volume compression, and resulting in a change in shape^[22]. During the cooling process, the t-ZrO₂ prones to changing into the m-ZrO₂, leading to a volume expansion of ZrO₂, while the expansion is constrained by Al₂O₃. Owing to the different CTEs between the ceramic reinforcement and metal matrix, thermal residual stress is produced at the interface during solidification^[23]. Thus, the limiting effect of Al₂O₃ on t-ZrO₂ is diminished, potentially leading to the initiation of the phase transition from t-ZrO₂ to m-ZrO₂. This phase transition of ZrO₂ in ZTA can decrease the thermal stress between the particles and matrix^[24].

3.2 Thermal expansion behavior of composites

The strain-temperature curves of the composites after the heating-cooling cycling process are depicted in Fig. 4, from which it can be seen that the length of the composite samples basically remains stable after the heat-cooling cycle, indicating a good dimensional stability of the prepared ZTA_p/HCCI composites in this study. In addition, fluctuations and deceleration in the deformation curves are observed when the temperature increases to 650–700 °C and 800–850 °C, which potentially attributed to the phase transformation within the ZTA ceramic particles accompanied by the volume shrinkage phenomenon^[22, 25].

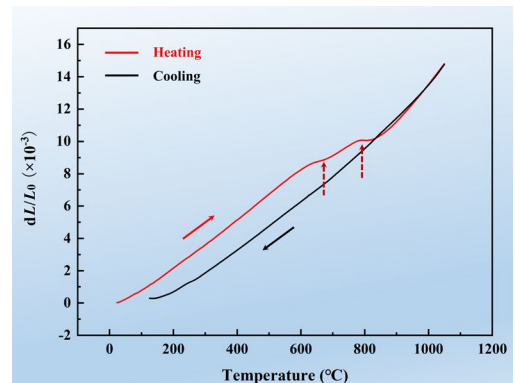


Fig. 4: Strain-temperature curve of composites during heating-cooling cycle

It has been shown that a lower interfacial bonding strength between the matrix and reinforcement can lead to the composites exhibiting significant residual strain after heating and cooling cycles^[26]. Shu et al.^[27] investigated the thermal expansion behavior of Cu matrix composites reinforced by SiC particles with and without Cu coating, and the results showed that the composites exhibited positive residual strains of 0.1%–0.3% when it reinforced with uncoated SiC particles. This is attributed to the insufficient strength of the weak interfacial bonding, which is unable to provide sufficient stress to restore the matrix to its initial dimensions during cooling after the composites experienced substantial plastic deformation in the heating phase. In contrast, no residual strain is found in the thermal cycling curve for the composites reinforced by SiC particles with coatings, indicating that the high interfacial strength reduces the internal residual strain of the composites. Zhang et al.^[28] studied the thermal expansion performances of Cu matrix composites reinforced with TiB₂ particles and found that the thermal expansion coefficient of the composites containing copper-coated particles was significantly reduced compared to that of the materials with uncoated particles, indicating that the interface of coated-particles and metal matrix exhibited higher bonding strength. Wang et al.^[29] also reported that the residual strain of the composites after thermal cycling was affected by interfacial bonding strength, and the strain decreased with increasing bonding strength. Therefore, the absence of residual strain in the thermal cycling curve shown in Fig. 4 can also further indicate the higher interfacial bonding strength between the particles and the matrix in this study.

In Fig. 5, the thermal expansion coefficient curves of the prepared ZTA/HCCI composites are presented, meanwhile, the instantaneous and average thermal expansion coefficients of the materials at various temperatures are also given. The average thermal expansion coefficient of the composites is $12.77 \times 10^{-6} \text{ K}^{-1}$, representing a 21.6% decrease compared to the HCCI materials with a coefficient of $16.3 \times 10^{-6} \text{ K}^{-1}$ (at 100–1,000 °C)^[30]. During the initial heating phase (25–200 °C), a notable increase in the CTE of the composites is observed as temperature rises, possibly attributed to the fact that after

the initiation of the heating process, sufficient time is required to achieve temperature homogenization inside the furnace^[31]. With the temperature continues to increase at 200–1,050 °C, except for the smaller fluctuations with a decreasing trend between 650–700 °C and 800–850 °C, there are no noticeable changes occurred for the composites CTE, which is fluctuating within the range of 11.79×10^{-6} – $14.42 \times 10^{-6} \text{ K}^{-1}$, indicating the relatively higher interfacial bonding strength, and the ceramic particles keep a higher constraint effect on the metal matrix during the heating process. The slight fluctuations with a decreasing trend during 650–700 °C and 800–850 °C correspond to the decrease in deformation at the corresponding temperatures in Fig. 4, which likely to be attributed to phase transitions within the ZTA particles, as mentioned earlier.

The thermal expansion characteristics of prepared composites are primarily influenced by the expansion behaviors of the ZTA reinforcing phase and the iron matrix together. Although both the iron matrix and the ZTA reinforcing phase expand with increasing temperature, the lower CTE of ZTA compared to the matrix imposes a constraining effect on the matrix expansion. Therefore, the addition of ZTA particles exerts a restraining and inhibiting effect on the matrix expansion, consequently leading to a decrease in the CTE of the composites. Additionally, during the heating phase of the thermal cycle, in the case of favorable interfacial bonding between the ZTA and the HCCI matrix, the ZTA produces tensile stress while the iron matrix generates compressive stress owing to the fact that the metal expansion is greater than that of the ceramics. Conversely, during the cooling stage, the iron matrix undergoes tensile stress, potentially enlarging the distance between Fe atoms and thus reduce the expansion capacity of the matrix. Meanwhile, the ZTA particles generate compressive stress that enhances their restriction effect on the matrix, consequently lessening the CTE of the composites.

The expansion of composites consists of two parts: expansion resulting from the rule of mixtures (ROM) and expansion originating from internal stress^[32]. The CTE stemming from internal stress is associated with the residual stress present in the matrix during heating and the average internal stress inside the matrix. Meanwhile, the thermal expansion characteristics of composites are influenced by both reinforcing phase and matrix phase, and the internal stresses generated by the differences in their CTE have the potential to change the overall CTE of the composites^[33]. Consequently, the current reduction in CTE is attributed to the results of ROM and the intense constraining effect of the ZTA on the HCCI matrix.

3.3 Theoretical calculation of CTE for composites

For the composites with known effective modulus of elasticity, the CTE can be achieved on the basis of determined relationship, whether using theoretical or experimental methods^[34]. Currently, various theoretical models have been developed to predict the CTE of particle-reinforced composites, primarily involving the rule of mixture (ROM),

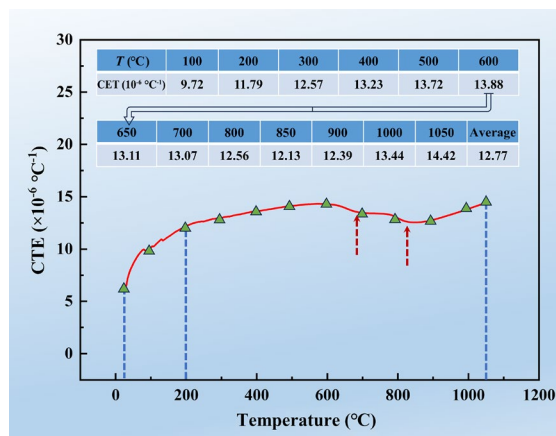


Fig. 5: Thermal expansion coefficient curve of composites

Turner, and Kerner models^[35]. Among them, the ROM model considered the relationship between the CTE of composites and the parameters of the reinforced and matrix phases is in accordance with the law of mixing, and the CTE can be calculated through the weighted mean value of the different components^[36]:

$$\alpha_c = \alpha_m \cdot f_m + \alpha_r \cdot f_r \quad (3)$$

where α_c , α_m , α_r represent the CTE of the composites, matrix, and reinforcement, respectively; f_m , f_r indicate the volume fractions of the matrix and reinforcement, respectively. Although the simplicity of the ROM in calculating the CTE, it overlooks the interaction among different components, which always presents between the two phases, and the thermal expansion property of the composites tends to be affected by the existed stress in interface.

In view of the above limitation, the Turner model was proposed, which considers the stress interaction among different phases. Assuming that the deformation of each component remains uniform during temperature variations, and that the stress generated inside the composites is positive (tensile or compressive), then the CTE for the composites can be obtained as shown below^[37]:

$$\alpha_c = \frac{\alpha_m K_m f_m + \alpha_r K_r f_r}{K_m f_m + K_r f_r} \quad (4)$$

where K_m and K_r are the bulk modulus of the matrix and reinforcement, respectively. However,

this is not limited to positive stresses between the different components. Therefore, Kerner introduced a model that accounts for the presence of positive stress and shear stress between the components^[38]. This model assumes that the reinforcing phase is spherical, discontinuous, and surrounded by a homogeneous matrix, which predicts the CTE of the composites as displayed below:

$$\alpha_c = \alpha_m f_m + \alpha_r f_r + (\alpha_r - \alpha_m) f_m f_r \times \frac{K_r - K_m}{K_r f_r + K_m f_m + \frac{3K_r K_m}{4G_m}} \quad (5)$$

where G_m represents the shear modulus of the matrix. The bulk modulus and shear modulus can be expressed using the following equations:

$$K_m = \frac{E}{3(1-2\mu)} \quad (6)$$

$$G_m = \frac{E}{2(1+\mu)} \quad (7)$$

where E represents the elastic modulus of the material, μ denotes the Poisson's ratio of the material. The aforementioned three models are all based on the assumption that both the reinforcing phase and the matrix metal exhibit linearly elastic strain, with the volume deformation of the material is considered to be small.

In this study, the fundamental performance parameters of the matrix HCCI and reinforcing phase ZTA are outlined in Table 1, where the elastic modulus and CTE of HCCI are taken as $E=177$ GPa (20–1,000 °C) and $\alpha=16.3 \times 10^{-6}$ K⁻¹ (100–1,000 °C), and these parameters for ZTA are determined as $E=260$ GPa and $\alpha=8.0 \times 10^{-6}$ K⁻¹. Subsequently, the provided data are substituted into the above three models, and the predicted values of the CTE for prepared composites based on various models can be obtained. Meanwhile, the comparative results between the predicted and tested values are shown in Table 2, revealing that the error is less than 2%.

Table 1: Performance parameters of different components in composites

	Elastic modulus (GPa)	Bulk modulus (GPa)	Shear modulus (GPa)	CTE (10 ⁻⁶ ·K ⁻¹)	Poisson's ratio
HCCI	177	147.5	68.1	16.3	0.3
ZTA	260	180.6	103.2	8.0	0.26

As shown from the above results, the calculated values for various models deviate from the experimental values. Specifically, the value calculated by the ROM model is larger than the experimental value, whereas the experimental value falls between the calculated values of the Turner and Kerner model and are closer to the calculated results through the Kerner model. This is consistence with the results of Sun et al.^[39] and Brooksbank et al.^[40], who found that the CTE of the particle-reinforced metal matrix composites typically falls between the calculated values of the Turner and Kerner models. With the increase in temperature, the contrasting CTE values of ZTA and HCCI result in mutual constraint within the composites, leading to the generation of complex stress inside the composites. The complex internal stresses, in turn, inhibit or promote the further

Table 2: Predicted values by different models and experimental value of CTE for composites

	Predicted values (10 ⁻⁶ ·K ⁻¹)			Experimental value (10 ⁻⁶ ·K ⁻¹)
	ROM model	Turner model	Kerner model	
ZTA/HCCI	12.98	12.56	12.83	12.77

expansion of ZTA and HCCI, and could potentially induce elastic-plastic deformation. Consequently, there exists a deviation between the experimentally measured CTE of the composite and its theoretically predicted value.

3.4 Effect of thermal expansion of composites on interfacial residual stress

It is generally recognized that the CTE of composites reflects the level of mismatch stress at the interface due to the addition of the reinforced phase, that is, the residual stress at the interface, which is mainly caused

by the CTE differences of the various components in the composites^[41, 42]. During the heating process, the expansion of the metal matrix is higher than that of the ceramic reinforcement, and the residual stress emerges as tensile stress on the ZTA particles and compressive stress on the iron metal, then results in the “thermal mismatch” and further leads to the generation of thermal stress at the interface.

Thermoplastic analysis can be carried out to investigate the thermal stress levels at the interface. According to the theories proposed by Brooksbank^[40] and Vaidya^[35], a model used in this study is shown in Fig. 6. It can be seen that an elastic spherical ceramic particle is inlaid in a spherical metal matrix, thus two types of stresses include radial stress σ_{rm} and circumferential stress $\sigma_{\theta m}$ exist on the metal side of the interface. These stresses can be mathematically formulated as following equations:

$$\sigma_{rm} = \frac{P[(r^3/R^3) - f_p]}{(1 - f_p)} \quad (8)$$

$$\sigma_{\theta m} = \frac{P[0.5(r^3/R^3) + f_p]}{(1 - f_p)} \quad (9)$$

$$P = \frac{(\alpha_m - \alpha_p)\Delta T}{[1.5 + 2.5\mu_m/E_m](1 - f_p) + [f_p(1 - 2\mu_p)/E_p]} \quad (10)$$

where r signifies the radius of the ceramic particle, R denotes the distance between the matrix and the center of the particle ($R \geq r$), f_p , μ , E stand for the volume fraction, Poisson's ratio, and Young's modulus (the subscripts p and m correspond to the particle and the matrix), ΔT reflects the temperature difference, and P symbolizes the interfacial pressure. It can be derived from the relationship among σ_{rm} , $\sigma_{\theta m}$, and P that the stress σ increases with decreasing R , and the interfacial stress reaches the maximum value when R decreases to r , accordingly, the plastic deformation of the matrix is most likely originates at the interface.

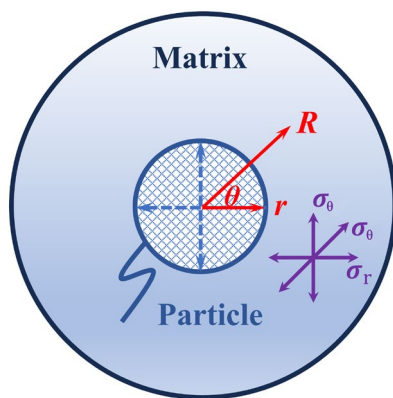


Fig. 6: Thermal stress model at interface for particle reinforced composites

The difference between σ_{rm} and $\sigma_{\theta m}$ can be expressed by:

$$\Delta\sigma = \sigma_{rm} - \sigma_{\theta m} = \frac{P[0.5(r^3/R^3) - 2f_p]}{(1 - f_p)} \quad (11)$$

when $\Delta\sigma = |\sigma_{rm} - \sigma_{\theta m}| > \sigma_y$ (σ_y represents the yield stress of the matrix at a given temperature), plastic deformation would be evoked within the matrix surrounding the interface^[43]. This phenomenon can be attributed to the Tresca yield criterion, which states that plastic deformation occurs in the matrix when the effective reduced shear stress exceeds the matrix yield stress^[44].

The values of interfacial stress P as well as stress difference $\Delta\sigma$ at varying temperatures for the ZTA/HCCI composites can be determined by bringing the relevant data in this study into Eqs. (10) and (11), respectively, as shown in Table 3. The yield stress of the HCCI matrix is taken as $\sigma_y = 340$ MPa at room temperature, this value decreases gradually with increasing temperature, and when the temperature increases to 700 °C, 800 °C, and 900 °C, σ_y decreases to 175 MPa, 110 MPa, and 63 MPa, respectively.

It can be seen from Table 3 that the value of σ is lower, whereas the yield stress of matrix σ_y is higher at lower temperatures, at which time the stress generated at the interface is insufficient to induce plastic deformation in the matrix. When the temperature exceeds 800 °C, the strength of matrix decreases and the thermal stress at the interface increases, until the condition $\sigma > \sigma_y$ is satisfied, then, the matrix surrounding the interface would experience plastic deformation, which corresponds to the CTE fluctuation around 800 °C in Fig. 5.

3.5 Mechanical property of composites through nanoindentation test

The positions of various phases for nano-indentation test are exhibited in Fig. 7(a), where four points are designated within ZTA and metal regions, three points are selected at the interface, then the average value is taken as the results for various areas. Moreover, the representative load-displacement (P - h) curves for ZTA, interface, and matrix are shown in Fig. 7(b), which is generated through recording the applied forces and corresponding displacements during the indenting process.

The hardness (H) and Young's modulus of different phases in the composites were calculated based on the “Oliver-Pharr” method^[45], with the relationships defined as:

$$H = \frac{P_{\max}}{A_c} \quad (12)$$

$$\frac{1}{E_r} = \frac{(1 - \nu^2)}{E} + \frac{(1 - \nu_i^2)}{E_i} \quad (13)$$

where $P_{\max}$ represents the maximum load applied during

Table 3: Calculation results of interfacial pressure P and stress difference $\Delta\sigma$ of composites at different temperatures

Temperature (°C)	100	200	300	400	500	600	700	800	900	1,000
P (MPa)	30.03	70.07	110.11	150.14	190.18	230.22	270.26	310.30	350.34	390.38
$\Delta\sigma$ (MPa)	15.02	35.04	55.06	75.07	95.09	115.11	135.13	155.15	175.17	195.19

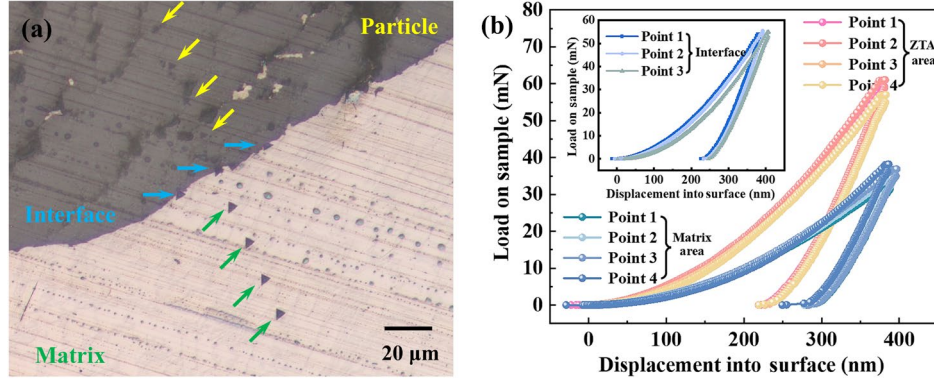


Fig. 7: Schematic representation of the indentation profile (a) and schematic of a typical load-displacement (P - h) relationship curves (b)

indentation test, A_c denotes the projected contact area of the indentation contour remaining on the sample surface, as depicted in Fig. 7(a). E_r signifies the equivalent elasticity modulus, E , E_i are the Young's modulus of the sample and indenter, respectively, and ν , ν_i represent the Poisson's ratios of the sample and indenter, respectively.

According to the results calculated using Eqs. (12) and (13), the indentation responses of the composites are illustrated in Fig. 8, from which it can be seen that the average hardness of the ZTA, interface, and matrix are 22.41 GPa, 13.2 GPa, and 10.89 GPa, respectively. The relatively lower hardness of the matrix compared to the interface and ZTA particles can be attributed to the close-rowed structure of the metal, allowing the atomic layers to slide under external forces without breaking the metallic bonds, resulting in enhanced toughness and reduced hardness^[46]. In addition, the hardness of the interface is between that of the ZTA and matrix, providing a transitional role. As the Young's modulus increases, the tendency of deformation decreases^[47]. The average modulus values for the ZTA, interface, and matrix are 377.8 GPa, 261.31 GPa, and 270.28 GPa, respectively. The ceramic phase, with the highest modulus, serves as the primary wear-resistant phase in the composites, protecting the metal matrix from damage during service. Meanwhile, the modulus of interface is smaller than that of the matrix, which can be attributed to the fact that the interface is formed through mechanical bonding. The interface with lower modulus tends to undergo plastic deformation, accommodating the phase strain of ZTA to some extent and reducing stress concentration around particles, thereby diminishing residual deformation and improving material properties, as previously reported by Vidilli et al^[48].

The total strain recovery ϵ_r and superplastic strain recovery ϵ_{sr} can be determined through following equations:

$$\epsilon_r = \frac{h_{\max} - h_f}{h_{\max}} \quad (14)$$

$$\epsilon_{sr} = \frac{h_{sr}}{h_{\max}} = \frac{h_{\max} - h_f - P_{\max}/S}{h_{\max}} \quad (15)$$

where $h_{\max}$ represents the depth under peak load $P_{\max}$, h_f denotes the residual depth after unloading, $h_{\max} - h_f$ signifies the recovery displacement, and S denotes the startup unloading slope. Figure 9 illustrates the values of ϵ_r and ϵ_{sr} for various

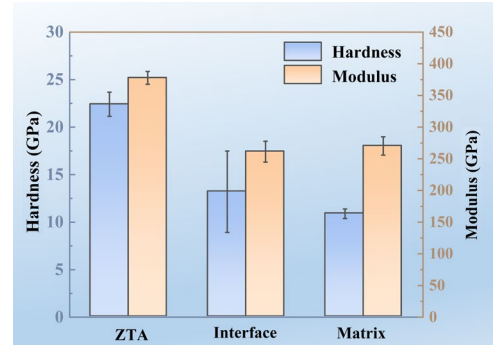


Fig. 8: Nanoindentation hardness and Young's modulus of composites

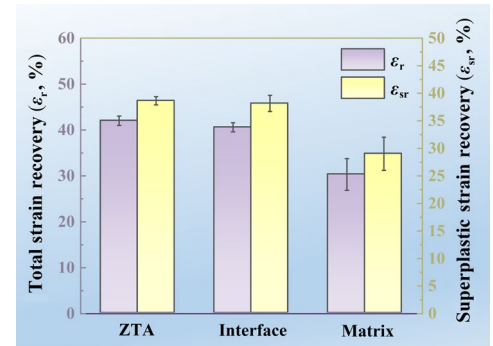


Fig. 9: Results of ϵ_r and ϵ_{sr} within composites

phases within the composites, where the ϵ_r values of the ZTA, interface, and HCCI are 42.02%, 40.58%, and 30.31%, respectively. Additionally, the ϵ_{sr} values for the ZTA, interface, and HCCI are 38.63%, 38.16%, and 29.01%, respectively. The findings indicate that the plastic deformation of the interface is close to that of the ZTA and lower than that of the matrix. Moreover, the elastoplastic behavior of the interface resembles that of the ZTA considering the comparable value of ϵ_r and ϵ_{sr} for the ZTA and interface.

4 Conclusions

In this work, ZTA particles reinforced HCCI matrix composites were prepared through a method termed flow mixing followed by pressure bonding, which without the need of pre-coating of the particles and the usage of preforms beforehand. The

microstructure, thermal expansion behavior, and mechanical properties of the composites were investigated and the following conclusions can be reached:

(1) The proposed technology is suitable for fabricating the particle-reinforced metal composites, and the results demonstrating a relatively uniform distribution of particles inside the metal matrix. Although the interface exhibits mechanical bonding, tight interfacial contact and high bonding strength are achieved. Furthermore, the fine grain size is obtained in the composites due to the exertion of a higher pressure. The composites primarily consist of Al_2O_3 , m-ZrO₂, t-ZrO₂, (Fe, Cr)₃C₂, γ -Fe, and α -Fe.

(2) The composites exhibit good dimensional stability during the heating and cooling processes in the temperature range of 20–1,050 °C, with an experimentally determined average CTE of $12.77 \times 10^{-6} \text{ K}^{-1}$. A comparison between experimental and theoretically calculated values of the CTE reveals that the error is less than 2%.

(3) The mechanical properties of the composites were evaluated through nanoindentation testing. The hardness of the ZTA, interface, and matrix follows the relationship of ZTA>interface>matrix, while the Young's modulus obeys ZTA>matrix>interface. Moreover, the strain recovery of various phases shows that the ZTA particles exhibit similar values to the interface and lower values than the matrix, indicating comparable elastic-plastic behavior between the interface and ZTA.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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