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Study on the interfacial structure of in-situ Al_2O_3 reinforced low-density iron-based metal matrix composite

Sai Chen [a], Shuangjie Chu [a,b,*], Bo Mao [a,*]

[a] *School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai 200240, China*
[b] *Baoshan Iron & Steel Cooperation Limited, Shanghai 201900, China*

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ABSTRACT

Lightweight high-aluminum steels suffer from a reduction in elastic modulus and limited performance, which restricts their structural applications. To address this challenge, we developed an in-situ approach to fabricate Al_2O_3-reinforced low-density iron-based metal matrix composites (IMMCs) by introducing CuO into molten high-Al steel. The in-situ reaction generated uniformly distributed Al_2O_3 particles, avoiding the dispersion and bonding issues associated with ex-situ methods. Microstructural characterizations revealed a graded interfacial layer with Fe, Al, O, and minor Cu, which provided strong metallurgical bonding between the reinforcement and the matrix. High-resolution TEM confirmed nanoscale lattice continuity across the interface, while EELS mapping and line scans demonstrated gradual compositional transitions that mitigated stress concentrations. Furthermore, ab initio molecular dynamics (AIMD) simulations elucidated the atomic-scale reaction pathway of Al_2O_3 formation. These results provide new insights into a scalable strategy for fabricating high-performance, low-density steels.

1. Introduction

High-aluminum low-density steels are gaining interest as structural materials due to their relatively low cost and high specific strength among the community of lightweight steels [1–3]. However, the high Al content, while reducing density, tends to lower the elastic modulus and degrade engineering performance and processing properties [4–6]. A promising solution to address this challenge is the introduction of ceramic reinforcements to counter the loss of elastic modulus [7,8]. Al_2O_3 is inexpensive, thermally stable, and already widely used as a deoxidizer in steel metallurgy, making it highly promising as an reinforcement phase for the development of IMMCs [9].

Conventionally, Al_2O_3 is introduced through external addition or powder metallurgy. However, current fabrication routes are difficult to scale up and often yield uneven reinforcement dispersion [10]. More critically, these ex-situ methods typically result in poor mechanical bonding between particles and matrices, leading to inferior interface strength and limited load transfer capability [11,12].

In this work, we report a novel in-situ reaction casting method in which CuO is added into molten high-Al iron-based melts to generate Al_2O_3. A gradient interfacial transition layer was observed between the Al_2O_3 and the matrix. The interfacial characteristics in terms of elemental distribution, crystal structure, and chemical bonding were investigated.

2. Methods

In this study, the Fe-10 wt.% Al-x wt.% CuO system was used to fabricate IMMCs consisting of α-Fe matrix and α-Al_2O_3 reinforcements through in-situ casting methods ($x = 4$ wt.%, 7 wt.%, 10 wt.%, 15 wt.%). The raw materials used were pure Fe and Al blocks and a mechanically alloyed powder mixture of Fe, Al, and CuO, as shown in Fig. S1. The detailed fabrication methods are shown in Fig. S2. Microstructural characterization was conducted using SEM, EDS, TEM, TKD, HRTEM, and EELS, with a detailed description in Supplementary Note 2. Thermodynamic calculations were carried out using FactSage to optimize the experimental parameters, which were discussed in Supplementary Note 3. AIMD simulations were performed to investigate atomic-scale interfacial structures and bonding characteristics, as described in Supplementary Note 4.

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* Corresponding authors.
E-mail addresses: sjchu@baosteel.com (S. Chu), bmao@sjtu.edu.cn (B. Mao).

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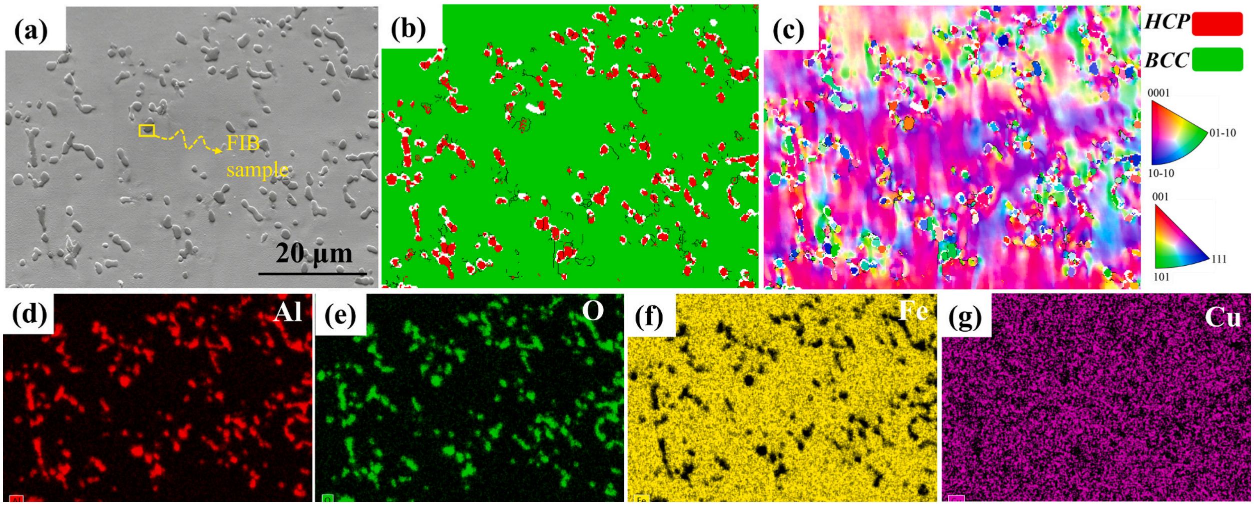

Fig. 1. (a) SEM image showing the distribution of Al_2O_3 in the IMMC with 10 wt% CuO addition, (b) phase map and (c) inverse pole figure (IPF) of IMMC sample, (d-g) EDS elemental maps of Fe, Cu, Al, and O, respectively.

3. Results and discussion

The microstructure of the IMMC sample with 10 wt% CuO addition is shown in Fig. 1. As shown in Figs. 1(a) and (b), the matrix exhibits BCC phase, with uniformly dispersed HCP phases distributed throughout. Combined with the analysis in Fig. 1(d-g), the BCC phase was determined to constitute the α-Fe, while the HCP phase is identified as Al_2O_3. The composite consists of 83.5 % α-Fe-based matrix and 16.5 % Al_2O_3 reinforcement, with the Al_2O_3 particles exhibiting an average size of 1.4 μm. The microstructural images and size distribution of the reinforcing phases for the other samples are presented in Supplementary Note 2. This result confirms the feasibility of fabricating in-situ Al_2O_3-reinforced high-Al iron-based composites via the traditional casting method. In this study, the interfacial characteristics between the in-situ Al_2O_3 and the matrix will be systematically investigated to elucidate the influence of the reaction mechanism on interfacial bonding.

Fig. 2(a) shows an SEM image of the FIB-milled sample, with Fig. 2(b, c) confirming the matrix is BCC ferrite and the Al_2O_3 phase is HCP. Fig. 2 (d) reveals an interface layer between Al_2O_3 and the matrix. EDS analysis shows elemental inter-diffusion at the interface. Fig. 2(e-h) display Fe, Cu, Al, and O maps, with Fe dominating the matrix. Al diffuses from the matrix, and O is released from CuO to form Al_2O_3. When reaction time or diffusion is limited, Cu accumulates at the interface, forming a compositional transition zone. This zone has a lower Cu content than the matrix and continuous gradients of Fe, Al, and O. The graded interfacial layer suggests a chemically integrated interface, potentially enhancing interfacial bonding strength. Fig. 2(i) shows the EELS scanning image, with the path marked. Fig. 2(j) quantifies the variation, confirming Al and O enrichment at the interface and slight Cu accumulation, indicating CuO involvement. Fig. 2(k) displays EELS elemental maps showing a nanoscale gradient, while Fig. 2(l) reveals HR-TEM lattice continuity across the interface, suggesting strong metallurgical bonding without amorphous layers. Fig. 2(m) presents SAED patterns showing two distinct diffractions: Fe-Al matrix and Al_2O_3, with interplanar spacings of ~ 0.2028 nm for the matrix and ~ 0.2378 nm for Al_2O_3. These findings confirm that in-situ Al_2O_3 forms a graded interfacial layer via metallurgical reactions [13,14]. The absence of amorphous phases, intermediate compounds, or defects, as confirmed by EELS and HR-TEM, indicates excellent interfacial bonding, and also reveals the evolution mechanism of the interface under in-situ reaction [15].

Fig. 2(i) shows a TEM image with an EELS scan path across the Fe-Al matrix and in-situ Al_2O_3 particle, analyzing 51 points along the interface. Fig. 3(a) presents low-loss spectra from six areas, showing increased energy loss toward Al_2O_3, consistent with its higher electron binding energy. Fig. 3 (b) shows the core-loss spectra which reveals a decrease in Fe and an increase in Al and O as the scan moves from the matrix to Al_2O_3. Areas 21–31 show coexistence of Fe, Al, O, and Cu, indicating a chemically graded interfacial zone. Pie charts display atomic ratios, confirming a diffusion-induced transition layer. EELS analysis reveals that the Al_2O_3-matrix interface forms through interdiffusion and reaction, promoting strong interfacial adhesion.

Fig. 4(a) illustrates the in-situ reaction mechanism for Al_2O_3 formation upon CuO addition to the Fe-Al melt. At high temperatures, CuO reacts with Al according to: $3CuO + 2Al \rightarrow Al_2O_3 + 3Cu$ ($\Delta G \approx -1856$ kJ/mol at 1550 °C), leading to the formation of Al_2O_3 particles. In the Fe-Al-CuO system, AIMD simulations at 1550 °C elucidate the in-situ formation mechanism of Al_2O_3 reinforcement. Initially, CuO retains its chain-like structure, but thermal perturbation causes Cu-O bonds to dissociate, releasing oxygen species (O^{2-}). These O atoms diffuse towards the Fe-Al matrix, where they interact with Al atoms, forming Al-O bonds and initiating the nucleation of Al_2O_3. This process is driven by atomic diffusion, bond exchange, and interfacial redox reactions. The interfacial evolution is governed by a complex interplay of reaction kinetics and thermodynamics. While the redox reaction is thermodynamically favorable, the limited reaction time leads to non-equilibrium conditions, causing the rapid formation of Al-O bonds. This results in a compositional gradient at the interface, with Al and Fe atoms participating in local bonding, and some Cu remaining within the interfacial region. The interface evolves into a self-organized gradient structure that minimizes interfacial energy, equalizes chemical potential, and alleviates strain.

4. Conclusions

In this study, dispersed Al_2O_3 reinforcement particles were successfully generated in a high-aluminum iron-based matrix via an in-situ reaction approach. SEM, EBSD, and HR-TEM combined with EELS analysis revealed that the interface between Al_2O_3 and the matrix is not a simple physical contact, but rather a graded transitional interfacial layer. This layer exhibits a distinct elemental gradient, where Fe, Al, and O inter-diffuse across the interface, indicating that the interface forms through a coupled diffusion–reaction mechanism. These findings offer new insights and theoretical support for the interfacial structure design of high-performance, low-density IMMCs.

CRediT authorship contribution statement

Sai Chen: Writing – original draft, Visualization, Data curation.

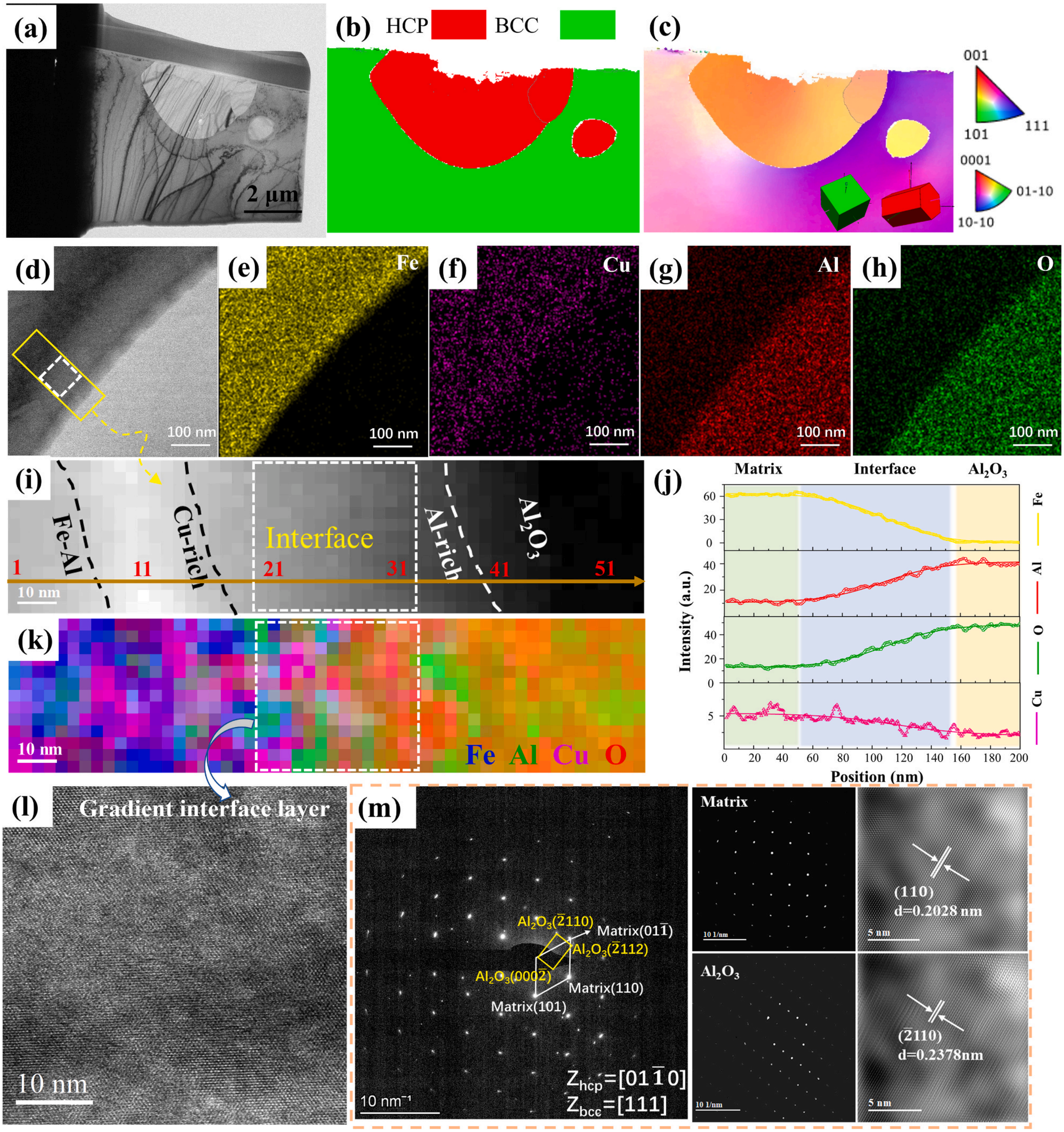

Fig. 2. (a) FIB image of the region marked in Figs. 1(a), (b) TKD phase map of the FIB sample, (c) IPF map corresponding to the same region, in which the crystal of the BCC matrix and HCP Al_2O_3 on the right is shown, (d) HAADF image showing the Al_2O_3 particle and matrix, and the interface between them, EDS maps illustrate the distribution of (e) Fe, (f) Al, (g) O, and (h) Cu within the composite, (i) EELS scan region, and (j) elemental distribution along the scan path indicated in (i), (k) EELS elemental spectrum, (l) HR-TEM image of the interface region, (m) SAED pattern of the graded interface, showing separated diffraction spots of the matrix and Al_2O_3, along with their respective interplanar spacings.

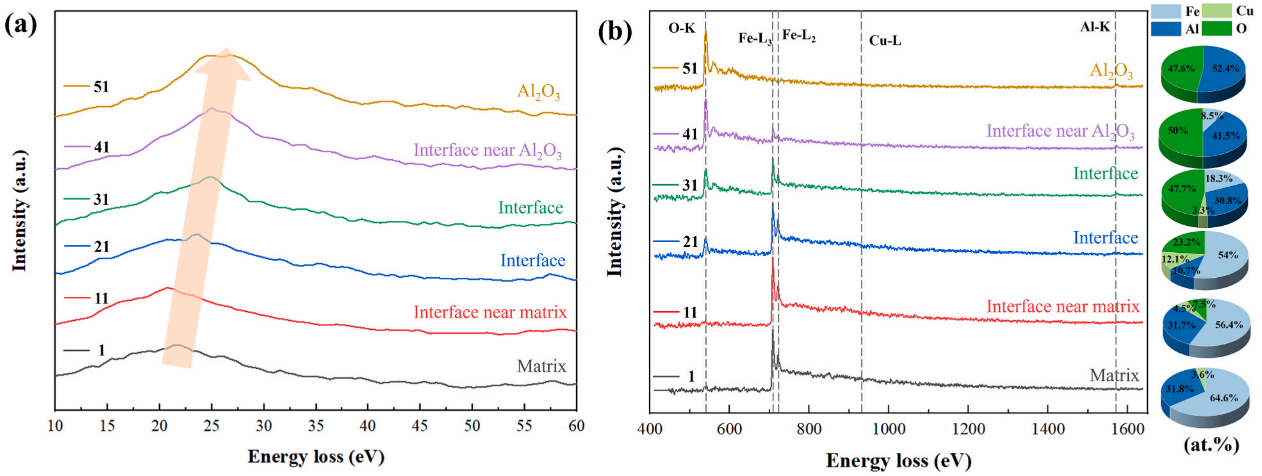

Fig. 3. (a) low-loss energy spectrum, and (b) high-loss energy spectrum and STEM-EDS of Fe, Al, Cu, and O showing the location of interasted area.

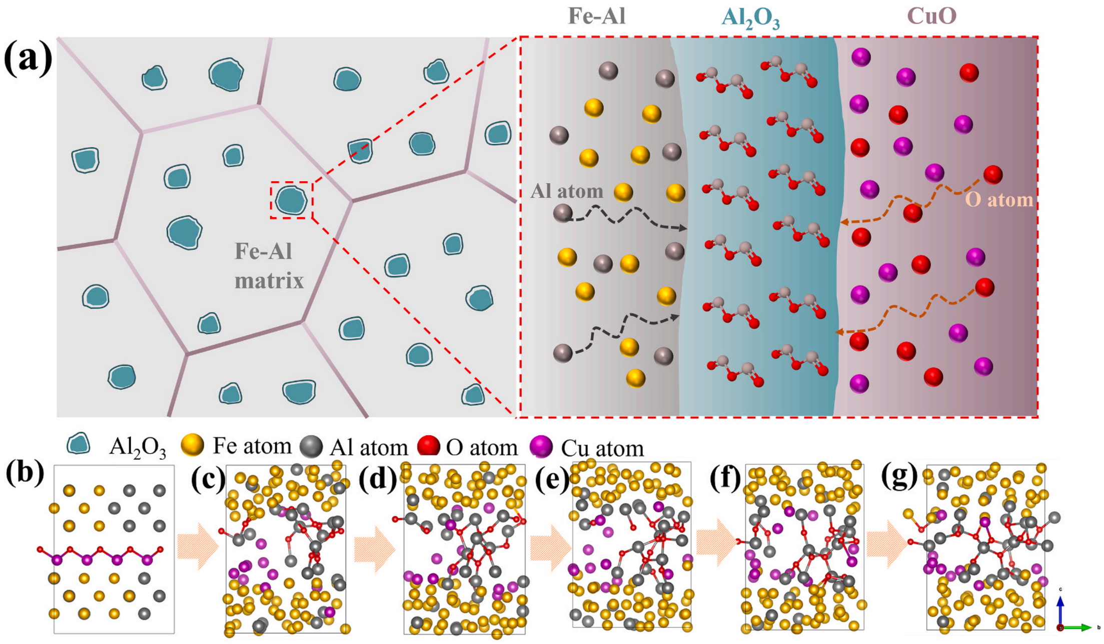

Fig. 4. (a) The diffusion of elements and the mechanism of in-situ reactions, (b-g) structural changes during high temperature relaxation of in situ reaction system.

Shuangjie Chu: Methodology, Investigation. **Bo Mao:** Writing – review & editing, Supervision, Project administration, Conceptualization.

Declaration of competing 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.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matlet.2025.139493.

Data availability

The data that has been used is confidential.

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