Fabrication of Bulk $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ Intermetallic Matrix Nanocomposite Through Mechanical Alloying and Sintering

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Abstract In the present study, $(\text{Fe,Cr})_3\text{Al}/20 \text{ vol\% } \text{Al}_2\text{O}_3$ nanocomposite was prepared through mechanochemical reactions during ball milling and successfully bulked using a combination of cold isostatic press and sintering at 1400 °C for 1 h. Two processing approaches were utilized to produce $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite: The first was milling of Fe, Cr, Al and Fe_2O_3 , while the second one was milling of Fe, Cr, Al and Cr_2O_3 , both in stoichiometric condition, to synthesize $(\text{Fe,Cr})_3\text{Al}/20 \text{ vol\% } \text{Al}_2\text{O}_3$. Structural changes of powder particles during mechanical alloying were studied by X-ray diffraction. The microstructure and the morphology of powder particles and bulk samples were also studied by scanning electron microscopy and transmission electron microscopy. Microstructural analysis showed that mechanochemical reactions took place during milling, and nanometric Al_2O_3 was uniformly distributed in the matrix. The results also showed that the second approach required a considerably higher milling time to produce $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite, as compared to the first one. For this reason, bulk samples were produced from the synthesized nanocomposite in the first approach. The microstructure of the sintered samples consisted of a network structure of $(\text{Fe,Cr})_3\text{Al}$ and Al_2O_3 phases with superior mechanical properties.

KEY WORDS: Iron aluminide; Mechanical alloying; Nanocomposite; Mechanochemical treatment

1 Introduction

Intermetallic compounds, as a new group of engineering materials, have been the focus of significant research and developments during the recent decades. Among them, iron aluminide intermetallic alloys with high strength, high resistance to oxidation and sulfidation, high-melting point, and low density show unique physical and mechanical properties [1, 2]. It should be noted that the absence of expensive elements like nickel on the iron aluminides

makes them cheaper than other high-temperature alloys. However, despite their attractive features, the application of iron aluminides has been limited by unsuitable ductility and toughness at the ambient temperature, as well as the loss of strength and creep resistance at temperatures higher than 600 °C [3]. Low ductility of intermetallics can be related to their ordered and low symmetry crystal structure with insufficient number of slip systems, which limits their plastic deformation. It has been shown that grain size refinement to nanometric scale can effectively improve the ductility of some intermetallic compounds [4, 5]. On the other hand, microstructural modification through the addition of second phase particles can be utilized to optimize the room temperature toughness of iron aluminides [6].

It has been shown that with the addition of hard second phase particles, such as TiC, TiB_2 and Al_2O_3 , both hardness and wear resistance of Fe_3Al can be significantly

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improved [7–9]. Al_2O_3 particles are well known as a suitable reinforcement for Fe_3Al matrix due to the close value of thermal expansion between these two phases. Also, they belong to a weak reaction system, so it can be expected that $\text{Fe}_3\text{Al}/\text{Al}_2\text{O}_3$ composite may show good physical and chemical stabilities at high temperatures [10]. Reinforcement particles can be added directly to the matrix (*ex situ* technique), or they can be produced through a mechanochemical reaction during the process (*in situ* reaction). Peng *et al.* [11] have shown that the *ex situ* added nano- Al_2O_3 particles to the Fe_3Al matrix cannot provide an effective nanocomposite with suitable mechanical properties. Severe agglomeration of second phase particles was introduced as the main reason for the unacceptable mechanical properties of *ex situ* synthesized $\text{Fe}_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite [11]. *In situ* formation of second phase particles in the intermetallic matrix has a lot of advantages such as the existence of strong chemical bonding between particles and the matrix and the lack of particles agglomeration, which can effectively increase the homogeneity of the final product [12–14]. There are many ways to produce $\text{Fe}_3\text{Al}/\text{Al}_2\text{O}_3$ composite. Casting has the advantage of low cost, but oxidation of the matrix at high temperatures and agglomeration of second phase particles are the main problems. Meanwhile, powder metallurgy technique has been successfully used to produce the bulk $\text{Fe}_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite [5, 6]. It has been shown that mechanical alloying (MA) of Fe_2O_3 and Al causes the formation of $\text{Fe}_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite with a homogenous distribution of second phase particles [5].

Many researchers have confirmed the positive effect of Cr on the mechanical properties of Fe_3Al intermetallic compound. The study by Sun *et al.* [15] confirmed the significant improvement of yield strength and creep resistance of Fe_3Al with 5% Cr addition. It was also reported [16] that the Cr addition could decrease hydrogen embrittlement in Fe_3Al . It has been proposed that the passive layer formed during the alloying of iron aluminides with Cr can effectively reduce the hydrogen reaction rate on the surface. Bai *et al.* [17] also showed that $\text{Fe}_3\text{Al}/\text{Al}_2\text{O}_3$ composite containing 4 wt% Cr exhibited excellent tribological properties at medium to high temperatures. Furthermore, corrosion resistance of the composite was significantly improved.

According to authors' knowledge, there is no published literature on the formation of the bulk $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite. Therefore, the aim of the present work was to produce the bulk $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite and study its microstructure and mechanical properties.

2 Materials and Methods

Fe (99.8% purity), Cr (99.5% purity), Al (99.5% purity), Cr_2O_3 (99.8% purity) and Fe_2O_3 (99.5% purity) powders were used as the starting materials. Figure 1 shows the SEM images of the as-received powder particles. Fe powders had nearly the same size of about 100 μm and a sponge morphology. Meanwhile, Cr particles were angular with the average size of 100–400 μm . Al powders were irregular in shape with the average particle size of 50–100 μm . As-received Cr_2O_3 particles were in an agglomerated condition, with fine particles size of less than 1 μm . Fe_2O_3 particles were also in the same agglomerated condition, with the average particle size of 1–4 μm .

Two approaches were employed to produce $(\text{Fe,Cr})_3\text{Al}$ –20 vol% Al_2O_3 nanocomposite, using mechanochemical reactions during ball milling. In the first approach, Fe, Cr, Al and Fe_2O_3 were mixed in a stoichiometric ratio and milled to conduct the following reaction:

In the second approach, Fe, Cr, Al and Cr_2O_3 were milled according to the following reaction:

It should be noted that in both cases, the stoichiometric ratio was chosen so that the final product would contain 20 vol% Al_2O_3 . Standard enthalpy change (ΔH_{298}°) and standard Gibbs free energy change (ΔG_{298}°) of reactions were calculated based on the available thermodynamic data in Table 1 [18]. The thermodynamic conditions for standard state were considered as the temperature of 298 K and the pressure of 1 atm (1.01×10^5 Pa).

Mechanical alloying was performed in a planetary ball mill machine under argon atmosphere and at the room temperature. Process controlling agent was used to prevent particles agglomeration during the milling process. Milling conditions in detail are summarized in Table 2. The milling was interrupted at selected time, and a small amount of powders was removed for further analysis. Compaction of the powder particles was performed using a cold isostatic press (CIP) under the pressure of 500 MPa. A rubber flexible mold was used in the CIP process. The sintering process was performed in an atmosphere-controlled furnace (Linn-HT1800G). The samples for cold isostatic press were sintered at 1400 °C for 1 h under argon atmosphere to produce dense bulk samples. Heating rate was chosen to be 10 °C/min. Bulk density measurements were taken using the Archimedes principle.

Fig. 1 Morphologies of the as-received powder particles: **a** Fe, **b** Cr, **c** Al, **d** Cr₂O₃, **e** Fe₂O₃

Table 1 Thermodynamic data used for the calculation of ΔH_{298}° and ΔG_{298}° of the reactions [18]

Compound	ΔH_{298}° (kJ/mol)	S_{298}° [kJ/(K mol)]	ΔG_{298}° (kJ/mol)
Fe	0	0.0273	−8.1
Cr	0	0.0236	−7
Al	0	0.0283	−8.4
Al ₂ O ₃	−1675.8	0.051	−1691
Cr ₂ O ₃	−1128.6	0.0811	−1152.8
Fe ₂ O ₃	−823.9	0.0874	−849.9

Table 2 Details of milling conditions

Rotation speed (rpm)	500
Diameter of vial (mm)	90
Capacity of vial (ml)	120
Vial material	Hardened chromium steel
Balls material	Hardened carbon steel
Diameter of balls (mm)	20
Ball to powders weight ratio	10:1
Total powder mass (g)	17

Phase transformations during MA and sintering were studied by X-ray diffraction (XRD, Philips XPERT MPD diffractometer utilizing Cu- K_{α} radiation). Crystallite size and the internal strain of the milled powders were obtained from the broadening of XRD peaks, using Williamson–Hall method [19].

$$\beta \cos \theta = (0.9\lambda/D) + 2\varepsilon \sin \theta. \quad (3)$$

where θ is the diffraction angle, D is the average crystallite size, λ is the wavelength of X-ray used and β is the peak width at the half-maximum intensity. The value of average strain can be estimated from the linear slope of $\beta \cos \theta$ versus $2 \sin \theta$, while the average crystallite size can be estimated from the intersection of this line at $\sin \theta = 0$.

The morphology and microstructure of the powder particles were studied by scanning electron microscopy (SEM, Philips XL30) and transmission electron microscopy (TEM, Philips EM208s). The hardness of the powder particles was determined using a microhardness tester with the load of 100 gr and the dwell time of 5 s. At least five measurements were carried out, and their averages were reported as the sample hardness.

3 Results and Discussions

Fe, Cr, Al and Fe₂O₃ powders were milled to produce (Fe,Cr)₃Al/Al₂O₃ nanocomposite according to reaction (1), which can be divided into two partial reactions:

where ΔH_{298}° and ΔG_{298}° for the reduction of Fe₂O₃ with Al (reaction 4) are about −852 and −840 kJ/mol [14], respectively. This shows that this reaction is exothermic and can thermodynamically occur during milling at room temperature.

There are two possible modalities for mechanochemical reactions to occur during the milling process [20]. One is sudden formation of the products through a combustion reaction, and the other one is gradual formation of the products with increasing the milling time.

It has been reported that the adiabatic temperature can be as a suitable criterion to predict the modality of mechanochemical reactions [20]. Adiabatic temperature is the highest temperature that a reaction system can reach. The value of the adiabatic temperature can be calculated using the following reaction:

$$\begin{aligned}
 -\Delta H_{298}^{\circ} = & \sum \left[n_p \int_{298}^{T_m^p} C_{ps}^p dT + n_d \int_{298}^{T_m^d} C_{ps}^d dT \right] \\
 & + \sum (n_p \Delta H_m^p + n_d \Delta H_m^d) \\
 & + \sum \left[n_p \int_{T_m^p}^{T_{ad}} C_{pl}^p dT + n_d \int_{T_m^d}^{T_{ad}} C_{pl}^d dT \right], \quad (6)
 \end{aligned}$$

where ΔH_{298}° is the enthalpy change at room temperature, n_p and n_d are the molar ratios, fractions of the products and the diluents, respectively, T_m^p and T_m^d are the melting points, C_{ps}^p and C_{ps}^d are the heat capacities in the solid phase, C_{pl}^p and C_{pl}^d are the heat capacities in the liquid phase, and ΔH_m^p and ΔH_m^d are the enthalpy of the fusion of the products and diluents, respectively. The second and third terms were included in Eq. (6) when melting of the products and the diluent phases occurred.

It has been reported empirically that combustion reaction can only occur when the adiabatic temperature is higher than 1800 K, implying that the modality of the mechanochemical reaction will be gradual if the adiabatic temperature is lower than 1800 K [4, 12]. Based on thermodynamic data in Ref. [18], the adiabatic temperature for reaction (4) was calculated to be 3381 K, showing that the reduction of Fe_2O_3 with Al should be a self-propagating combustion reaction. Meanwhile, the existence of 5 mol Fe, 3.5 mol Cr and 3.5 Al in the mixture as diluents caused the reduction of the adiabatic temperature to the 1563 K. So it was expected that reaction (1) would occur in a gradual mode.

Figure 2a shows the XRD patterns of Fe–Cr–Al– Fe_2O_3 powder mixtures during MA. Powder mixtures contained Fe, Cr, Al and Fe_2O_3 diffraction peaks after 1 h milling time. Overtime, the intensity of the as-received peaks was decreased. In addition, the (110) iron peak was displaced during the milling (Fig. 2b), which confirmed the partial Cr and Al were soluted into the iron lattice during MA. In other words, Fe(Cr,Al) solid solution was firstly formed in a gradual mode. Increasing the milling time to 20 h led to

the transformation of Fe(Cr,Al) solid solution to the $(\text{Fe,Cr})_3\text{Al}$ intermetallic compound, which was accompanied with the XRD peak position change (Fig. 2b). No sign of Fe_2O_3 was detected for milling of 20 h, showing the completion of the aluminothermic reaction between Fe_2O_3 and Al. The effect of Cr on the formation mechanism of $(\text{Fe,Cr})_3\text{Al}$ and the thermodynamic behavior of the Fe–Cr–Al ternary system could be found in our previous report [21]. It showed that the addition of Cr to the binary Fe–Al system decreased the Gibbs free energy; furthermore, $(\text{Fe,Cr})_3\text{Al}$ was found to be more stable than Fe_3Al . Further milling only caused the broadening of the $(\text{Fe,Cr})_3\text{Al}$ peaks. It should be noted that no sign of Al_2O_3 peak was detected in the synthesized nanocomposite. This could be due to the amorphous nature of the produced Al_2O_3 or its nanometric size. It has been reported that the amorphous or crystalline nature of in situ synthesized Al_2O_3 during MA depends on the modality of mechanochemical reactions. Normally the gradual proceeding of reduction process during mechanical alloying sustains relatively low temperature, which favors the formation of amorphous alumina [22]. Meanwhile, the heat generated from the sudden formation of the products through a combustion reaction can make the amorphous Al_2O_3 to be crystallized [23]. Low adiabatic temperature and gradual nature of reactions in the present powder mixture can be explained the main possible reasons for the synthesis of amorphous Al_2O_3 . Amorphous structure of in situ produced Al_2O_3 was previously confirmed by Rafiei *et al.* [24] in the $(\text{Fe,Ti})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite after gradual redox reaction during ball milling. All of these factors, together with the existence of the internal strain in the milled powders, can be regarded as the possible reasons for the lack of Al_2O_3 peaks in the nanocomposite after MA. The crystallite size and the internal strain of the $(\text{Fe,Cr})_3\text{Al}$ phase after 60 h of MA were 12 nm and 1.1%, respectively.

Figure 3 shows the bright field and dark field TEM micrographs and selected area diffraction (SAD) pattern (inset) of the $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite after 60 h milling. TEM micrographs could obviously confirm the existence of nanometric Al_2O_3 with the size of less than 10 nm. It should be noted that the Al_2O_3 reinforcements were homogeneously distributed in the intermetallic matrix. This could be regarded as one advantage of in situ synthesized nanocomposites, as compared to the ex situ ones. The uniform distribution of second phase particles produced through displacement reaction was previously reported [4–6]. TEM observation of in situ synthesized $(\text{Al}_2\text{O}_3\text{--Si})/\text{Al}$ nanocomposite by Mokhnache *et al.* [25] showed that the Al_2O_3 particles were uniformly distributed in Al matrix, and the interface was clean. The fine and homogenous distribution of second phase particles could

Fig. 2 XRD patterns of the Fe–Cr–Al–Fe₂O₃ powder mixture **a**, the magnified XRD peak around 2θ of 43° – 46° **b**

Fig. 3 TEM micrographs and SAD pattern (*inset*) of the 60 h milled (Fe,Cr)₃Al/Al₂O₃ nanocomposite produced through the MA of the Fe–Cr–Al–Fe₂O₃ powder mixture: **a** bright field image, **b** dark field image

effectively improve the mechanical properties of the nanocomposite.

Fe, Cr, Al and Cr_2O_3 powders were also milled to produce the $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite according to reaction (2). This reaction can be divided to the following reactions:

ΔH_{298}° and ΔG_{298}° for the reduction of Cr_2O_3 with Al (reaction 7) were about -547 and -535 kJ/mol, respectively [26]. Also, its adiabatic temperature was 2331 K. The addition of some diluents caused the decrease in the adiabatic temperature to 1279 K, which was lower than 1800 K, and predicted that reaction (2) would occur in a gradual mode.

Figure 4 shows the XRD patterns of the Fe–Cr–Al– Cr_2O_3 powder mixture during MA. With increasing the milling time up to 20 h, the only visible change was broadening of the initial peaks. $(\text{Fe,Cr})_3\text{Al}$ diffraction peaks were first observed after 40 h of milling, showing the occurrence of the mechanochemical reaction between Cr_2O_3 and Al. It should be noted that some Cr_2O_3 peaks were still present in the XRD pattern of 40 h milled powders, implying that the reaction was still incomplete. Further milling up to 100 h led to the completion of the mechanochemical reaction and the synthesis of $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite. As can be seen, the reaction completion during MA of Fe–Cr–Al– Cr_2O_3 required a considerably higher milling time, in comparison with Fe–Cr–Al– Fe_2O_3 . The reason could be the adiabatic temperature difference between these two alloying systems. The adiabatic temperature of the reaction (1) (Fe–Cr–Al– Fe_2O_3 powder mixture) was 1563 K, while it was 1279 K for reaction (2) (Fe–Cr–Al– Cr_2O_3 powder mixture). The lower adiabatic temperature during MA of the Fe–Cr–Al– Cr_2O_3 powder mixture caused the lower driving force for the completion of mechanochemical reactions during ball

milling. Bright field and dark field TEM micrographs of the 100 h milled powders (Fig. 5) showed the existence of nanometric Al_2O_3 reinforcement in the $(\text{Fe,Cr})_3\text{Al}$ matrix. The crystallite size and the internal strain of $(\text{Fe,Cr})_3\text{Al}$ phase after 100 h milling were 15 nm and 1.2%, respectively.

To make the comparisons between the $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite produced through the reduction of Fe_2O_3 and Cr_2O_3 , XRD patterns of these two nanocomposite were compared after annealing at 900 °C for 1 h, as shown in Fig. 6. As can be seen, the position of $(\text{Fe,Cr})_3\text{Al}$ and Al_2O_3 peaks was exactly the same, showing that the two mentioned approaches could produce the same nanocomposite. However, less time required for the formation of the nanocomposite through the reduction of Fe_2O_3 led to the use of this powder mixture for sintering and production of bulk samples.

Figure 7a shows the morphology of $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite after 60 h of milling. The powder particles contained agglomerates of very fine particles of about 1 μm in size. Embrittlement of the powder particles could be regarded as the main reason for the fragmentation of the particles during milling. Powder mixtures after 60 h MA were cold isostatic pressed and sintered to produce the bulk $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite. The sintering process was performed at 1400 °C for 1 h according to the literature [5, 27, 28]. The same sintering process allowed the comparison of the final product properties. Figure 7b shows typical examples of the bulk nanocomposite samples. The diameter and height of the samples were 14 and 18 mm, respectively. The remaining porosity, measured by Archimedes method, was below 7% for all samples.

The XRD patterns of the $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite after MA and the subsequent sintering are shown in Fig. 8. As can be seen, the positions of $(\text{Fe,Cr})_3\text{Al}$ diffraction peaks in the as-milled and as-sintered samples were the same and only peaks width was decreased, showing some degree of grain growth. In addition, Al_2O_3 peaks could be obviously observed in the XRD pattern after sintering. Table 3 shows the crystallite size and the internal strain of $(\text{Fe,Cr})_3\text{Al}$ and Al_2O_3 phases after MA and sintering. The sintering process caused the grain growth of $(\text{Fe,Cr})_3\text{Al}$ from 12 nm to 66 nm, and also, the reduction in its internal stain from 1.1 to 0.33%. The crystallite size of the in situ synthesized Al_2O_3 after the sintering process was about 42 nm. The slight increase in the crystallite size during the sintering process was predictable. It should be noted that the presence of nanometric Al_2O_3 particles could effectively retard grain growth through the grain boundary pinning effect during sintering [12].

SEM micrographs of the bulk $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite at two magnifications, along with EDS analysis of different phases, are shown in Fig. 9. It could be

Fig. 4 XRD patterns of the Fe–Cr–Al– Cr_2O_3 powder mixture during ball milling

Fig. 5 TEM micrographs and SAD pattern (*inset*) of the $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite produced through the MA of the $\text{Fe-Cr-Al-Cr}_2\text{O}_3$ powder mixture: **a** bright field image, **b** dark field image

Fig. 6 XRD patterns of the $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite produced through the reduction of Fe_2O_3 **a**, Cr_2O_3 **b** after annealing at 900°C for 1 h

Fig. 8 XRD patterns of the $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite before **a**, after **b** sintering at 1400°C for 1 h

Fig. 7 **a** Morphology of $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite after 60 h MA, **b** typical examples of the cold isostatic pressed nanocomposite

seen that the $(\text{Fe,Cr})_3\text{Al}$ and Al_2O_3 phases were homogeneously distributed in the bulk sample matrix. Some porosities were also visible in the microstructure. EDS analysis showed that the light gray and dark gray phases in

the microstructure were $(\text{Fe,Cr})_3\text{Al}$ and Al_2O_3 , respectively.

The final distribution of the phases in the microstructure confirmed that $(\text{Fe,Cr})_3\text{Al}$ and Al_2O_3 particles formed a

Table 3 Crystallite size and the internal strain of $(\text{Fe,Cr})_3\text{Al}$ and Al_2O_3 phases before and after sintering

Phase	Before sintering		After sintering	
	Crystallite size (nm)	Internal strain (%)	Crystallite size (nm)	Internal strain (%)
$(\text{Fe,Cr})_3\text{Al}$	12	1.1	66	0.33
Al_2O_3	–	–	42	0.29

Fig. 9 Microstructures of the bulk $(\text{Fe,Cr})_3\text{Al}/\text{Al}_2\text{O}_3$ nanocomposite at two magnifications **a**, **b**, the EDS analysis of light gray and dark gray regions **c**, **d**

network structure which finally grew up through the sintering diffusion process. The uniform distribution of the phases in the microstructure without the formation of “core-rim structure” is resulted from ball milling process. Similar microstructure was previously reported by Kim *et al.* [29] for the $(\text{Ti,Mo})\text{C}$ –Ni nanocomposite after sintering, which has been related to the extremely fine and homogenous nanocomposite powder obtained from mechanical alloying. Meanwhile, the microstructure of the $(\text{Fe,Cr})_3\text{Al}/20 \text{ vol}\% \text{ Al}_2\text{O}_3$ nanocomposite produced by Subramanian *et al.* [27, 28] through the reaction sintering

of $\text{Fe}/\text{Al}/\text{Fe}_2\text{O}_3$ was composed of Al_2O_3 rings around iron aluminide grains. This microstructure was related to the occurrence of reduction reaction at the interface of $\text{Fe}-40\text{Al}$ and Fe_2O_3 powders. It has been shown that the presence of the network microstructure in the intermetallic matrix composite arises from mechanochemical reaction during ball milling and indicates superior hardness [5].

Table 4 shows the microhardness of the $(\text{Fe,Cr})_3\text{Al}/20 \text{ vol}\% \text{ Al}_2\text{O}_3$ bulk sample produced in the present work and the comparison with the same composite produced through the other techniques. As can be seen, the microhardness of

Table 4 Microhardness value of the (Fe,Cr)₃Al/Al₂O₃ nanocomposite produced in the present work and the comparison with Fe₃Al/Al₂O₃ composite produced by other techniques

Composition	Synthesis method	Hardness (HV)
(Fe,Cr) ₃ Al–20 vol% Al ₂ O ₃	Present work	626 ± 40
Fe ₃ Al–20 vol% Al ₂ O ₃ [27]	Reaction sintering of Fe–40Al–Fe ₂ O ₃ followed by sintering at 1300 °C for 1 h	222
Fe ₃ Al–20 vol% Al ₂ O ₃ [28]	Reaction sintering of Fe–40Al–Fe ₂ O ₃ followed by sintering at 1300 °C for 1 h under the pressure of 35 MPa	355 ± 30
Fe–40Al–8Cr + 20 vol% Al ₂ O ₃ [28]	Reaction sintering of Fe–40Al–8Cr–Fe ₂ O ₃ followed by sintering at 1300 °C for 1 h under the pressure of 35 MPa	385 ± 33

the synthesized nanocomposite was 626 ± 40 HV, which was considerably higher than that of the others. Since all these composites had the same percentage of reinforcement phase, difference in their mechanical properties could be due to the distribution and size of the reinforcement phase. In the mechanochemically synthesized sample (present work), the (Fe,Cr)₃Al and Al₂O₃ phases formed a continuous fine network structure in micron or submicron size. On the other hand, the presence of nanometric Al₂O₃ particles in the (Fe,Cr)₃Al could effectively increase its hardness.

4 Conclusions

- (Fe,Cr)₃Al/Al₂O₃ nanocomposite was successfully synthesized through mechanochemical reactions during ball milling.
- It was observed that ball milling of Fe, Cr, Al and Fe₂O₃ could produce (Fe,Cr)₃Al/Al₂O₃ nanocomposite in less milling time, as compared to the ball milling of Fe, Cr, Al and Cr₂O₃.
- Microstructural analysis confirmed the presence of in situ synthesized nanometric Al₂O₃ homogenously distributed in the (Fe,Cr)₃Al matrix.
- CIP and sintering were used to produce bulk nanocomposite samples. It was observed that (Fe,Cr)₃Al and Al₂O₃ phases were homogenously distributed in a network microstructure.
- (Fe,Cr)₃Al/20 vol% Al₂O₃ bulk nanocomposite sample showed the average microhardness of 626 HV, which was about twice higher than that of the Fe₃Al/20 vol% Al₂O₃ composite prepared by other techniques.

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