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## IN SITU SYNTHESIS OF CHROMIUM-RICH NITRIDES IN A MECHANICALLY ACTIVATED Fe–FeCr POWDER SYSTEM

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synthesis, chromium  
nitride, nitrogen sintering,  
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### ABSTRACT

Three Fe–FeCr powders processed in a planetary centrifugal mill for 15, 30, and 60 min were evaluated as precursors for the in situ synthesis of Cr-rich nitrides. The initial mixture contained 82.03 wt.% reduced Fe powder and 17.97 wt.% low-carbon ferrochrome (LC FeCr), corresponding to a nominal Cr content of 13 wt.%. Particle morphology, size distribution, Cr redistribution, BCC structure, and cold compactability were examined using SEM, EDS, XRD, image analysis, and uniaxial pressing. During the first 30 min, plastic deformation and cold welding competed with particle fragmentation, resulting in temporary formation of coarse secondary agglomerates. After 60 min, work hardening promoted fracture and produced finer, predominantly irregular particles. From 15 to 60 min, D50 decreased from approximately 90 to 53  $\mu\text{m}$ , while the apparent BCC crystallite size decreased from 19.8 to 17.1 nm. These structural changes were accompanied by reduced green density and increased susceptibility to cracking. The 60-min compact was sintered at 1150 °C for 1 h in high-purity N<sub>2</sub>. Elemental mapping revealed N enrichment within Cr-rich regions. Their local composition was consistent with an M<sub>2</sub>N-type phase, provisionally identified as (Cr,Fe)<sub>2</sub>N. The results demonstrate that mechanical activation improves precursor dispersion and nitriding reactivity, although excessive processing adversely affects powder compactability.

### INTRODUCTION

In situ formation of nitride phases during consolidation offers a direct route to Fe-based composites because the reinforcing constituent is generated from the powder precursor rather than introduced as a separate ceramic powder. Mechanical activation can promote this route by refining the reactants, increasing interfacial area, and accumulating crystal defects that accelerate subsequent gas–solid reactions. The processing window must nevertheless be controlled because severe deformation also changes particle shape, cold-welding behavior, and compactability (Manohar et al., 2018; Akhmetov & Ereemeeva, 2022; Saxena et al., 2023).

LC FeCr is a practical Cr-bearing precursor for such reactions. Unlike high-carbon FeCr, which favors Cr carbide formation in Fe-based powder composites (Karaimchuk et al., 2020), its limited carbon content allows Cr-rich regions to react preferentially with N during heat treatment (Hu et al., 2021). Cr-rich nitrides are attractive reinforcing phases because of their hardness, wear resistance, chemical stability, and corrosion resistance (Li et al., 2006; Yeo et al., 2022).



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The use of LC FeCr fines also supports resource-efficient powder processing. Crushing and screening of commercial ferrochrome generate dust and undersized fractions that are difficult to recycle through conventional smelting routes (du Preez et al., 2023; Sariyev et al., 2025). Their direct use remains challenging because brittle fracture, compositional heterogeneity, inclusions, and surface oxidation influence powder refinement, Fe/FeCr contact development, compaction, and nitriding kinetics (Gasik, 2013; Kel' et al., 2025).

This study considered three Fe–FeCr precursor powders mechanically processed in a planetary centrifugal mill for 15, 30, and 60 min. The objective was to determine how processing time changes particle morphology, size distribution, Cr dispersion, BCC-domain structure, and green compactability, and to evaluate in situ formation of a Cr-rich nitride during nitrogen sintering. All three powders were compared by SEM-based particle analysis and pressing tests; phase and elemental-map comparisons were restricted to the available Fe–FeCr-15 and Fe–FeCr-60 datasets, and nitriding was evaluated for the selected Fe–FeCr-60 compact.

## MATERIALS AND METHODS

Reduced iron powder and low-carbon ferrochrome (LC FeCr) fines were used to prepare the Fe–FeCr reaction precursor. Both powders were passed through a 200- $\mu\text{m}$  sieve before blending. Their major-element compositions are listed in Table 1. The mixture contained 82.03 wt.% reduced iron powder and 17.97 wt.% LC FeCr, giving a nominal Cr concentration of 13 wt.%. No additional alloying or nitride-forming constituents were introduced. Before mechanical processing, the blend was dried at 120 °C for 60 min.

**Table 1.** Chemical compositions of the starting powders, wt.%

| Material            | Fe   | Cr    | Si   | C    | P    | S    |
|---------------------|------|-------|------|------|------|------|
| Reduced iron powder | bal. | -     | -    | 0.01 | 0.02 | 0.02 |
| LC FeCr             | bal. | 72.32 | 0.20 | 0.07 | 0.02 | 0.02 |

A 20.00-g powder charge and 200 g of grinding balls were loaded into a 100-mL steel vial, corresponding to a ball-to-powder mass ratio of 10:1. The vial and grinding media were made of AISI 304 stainless steel. Three powder batches, designated Fe–FeCr-15, Fe–FeCr-30, and Fe–FeCr-60, were mechanically processed in air in a planetary centrifugal mill (PCM) at 800 rpm for 15, 30, and 60 min, respectively. Wear of the milling assembly was considered negligible relative to the powder charge; its principal constituents, Fe and Cr, were also the major elements of the investigated system.

Each mechanically processed powder was consolidated by single-action uniaxial pressing in a cylindrical die with an inner diameter of 12 mm at 200–700 MPa. Each charge weighed 2.64 g. The charge mass was determined from  $m = \rho_{\text{th}} \cdot V \cdot k$ , where  $\rho_{\text{th}} = 7.8 \text{ g/cm}^3$  is the theoretical density of the Fe-based material,  $V$  is the volume of a cylinder 12 mm in diameter and 6 mm in height, and  $k = 0.5$  is the target relative green-density coefficient. The actual green density was calculated geometrically from the measured mass and specimen dimensions. For each powder condition and pressing pressure, three independently pressed compacts were measured ( $n = 3$ ). Green-density values are reported as the mean  $\pm$  standard deviation.

The Fe–FeCr-60 powder, which exhibited the most advanced particle refinement and Cr redistribution, was selected for the nitriding experiment. Its compacts were sintered at 1150 °C for 1 h in flowing high-purity  $\text{N}_2$  (99.999%). A gas flow rate of approximately 3000 mL/min was maintained during heating, isothermal holding, and furnace cooling to room temperature. Nitrogen was therefore supplied only during sintering, so any Cr–N-rich product detected afterward was formed in situ.

Particle morphology was examined for the Fe–FeCr-15, Fe–FeCr-30, and Fe–FeCr-60 powders using a ZepTools ZEM20 scanning electron microscope. For each condition, 250–300

particles were measured from SEM micrographs using ImageJ 1.54f; the arithmetic mean particle size, D50, D90, and interquartile range (D25–D75) were determined. Elemental distributions were characterized for the Fe–FeCr-15 and Fe–FeCr-60 powders and the sintered Fe–FeCr-60 compact using an Oxford Instruments energy-dispersive X-ray spectroscopy (EDS) system.

## RESULTS AND DISCUSSION

### Mechanical Activation of Fe–FeCr-15, Fe–FeCr-30, and Fe–FeCr-60 Powders

The Fe–FeCr-15 powder contained predominantly large flattened and lamellar Fe-rich particles, some approximately 200–250  $\mu\text{m}$  in size (Figure 1a). Plastic deformation and cold welding of ductile Fe therefore dominated the first 15 min of PCM processing. The smaller angular particles were mainly brittle LC FeCr fragments or pieces detached from mechanically bonded Fe–FeCr composite particles.

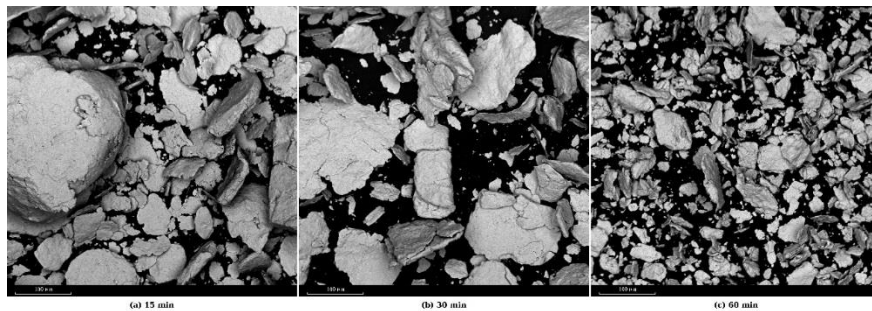


**Figure 1.** Morphology of Fe–FeCr powders mechanically processed in the PCM:  
(a) Fe–FeCr-15, (b) Fe–FeCr-30, and (c) Fe–FeCr-60

In the Fe–FeCr-30 powder, the lamellar fraction remained evident, while repeated cold welding produced a limited population of coarse secondary agglomerates. D90 increased to approximately 237  $\mu\text{m}$  even though D50 decreased relative to the Fe–FeCr-15 condition (Table 2). Thus, fine-particle accumulation and agglomerate growth occurred simultaneously. This mixed refinement–coarsening response is characteristic of early mechanical activation, when work hardening is not yet sufficient for fracture to remain dominant (El-Eskandarany, 2020).

**Table 2.** Particle-size characteristics of the mechanically processed Fe–FeCr powders

| Processing time, min                                                                | Mean size, $\mu\text{m}$ | D25, $\mu\text{m}$ | D50, $\mu\text{m}$ | D75, $\mu\text{m}$ | D90, $\mu\text{m}$ |
|-------------------------------------------------------------------------------------|--------------------------|--------------------|--------------------|--------------------|--------------------|
| 15                                                                                  | 108                      | 64                 | 90                 | 125                | 160                |
| 30                                                                                  | 103                      | 35                 | 65                 | 131                | 237                |
| 60                                                                                  | 70                       | 35                 | 53                 | 82                 | 140                |
| <i>Note – values are rounded to the nearest micrometer; prepared by the authors</i> |                          |                    |                    |                    |                    |

The Fe–FeCr-60 powder showed the most pronounced morphological change. Coarse lamellae had largely disappeared and were replaced by finer, irregular, near-equiaxed particles. D50 decreased to approximately 53  $\mu\text{m}$  and D90 to 140  $\mu\text{m}$ . Accumulated strain and work hardening reduced further flattening of Fe-rich particles, causing subsequent impacts to favor fracture. The 60-min state therefore provided the finest and most highly activated precursor for the subsequent in situ nitriding experiment.

### Evolution of the Cr-Rich Nitride Precursor

Elemental maps showed progressive refinement of the Cr-bearing precursor (Figure 2). After 15 min, Cr was concentrated in several large isolated ferrochrome regions, whereas Fe

occupied most of the mapped area. After 60 min, Cr-rich regions were smaller and more numerous, indicating fragmentation of ferrochrome and its incorporation into Fe-rich composite particles. Local variations in Cr intensity remained; the powder was therefore a mechanically activated heterogeneous precursor rather than a fully homogeneous prealloyed powder. This retained heterogeneity supplied localized Cr-rich sites for the subsequent gas–solid nitriding reaction.

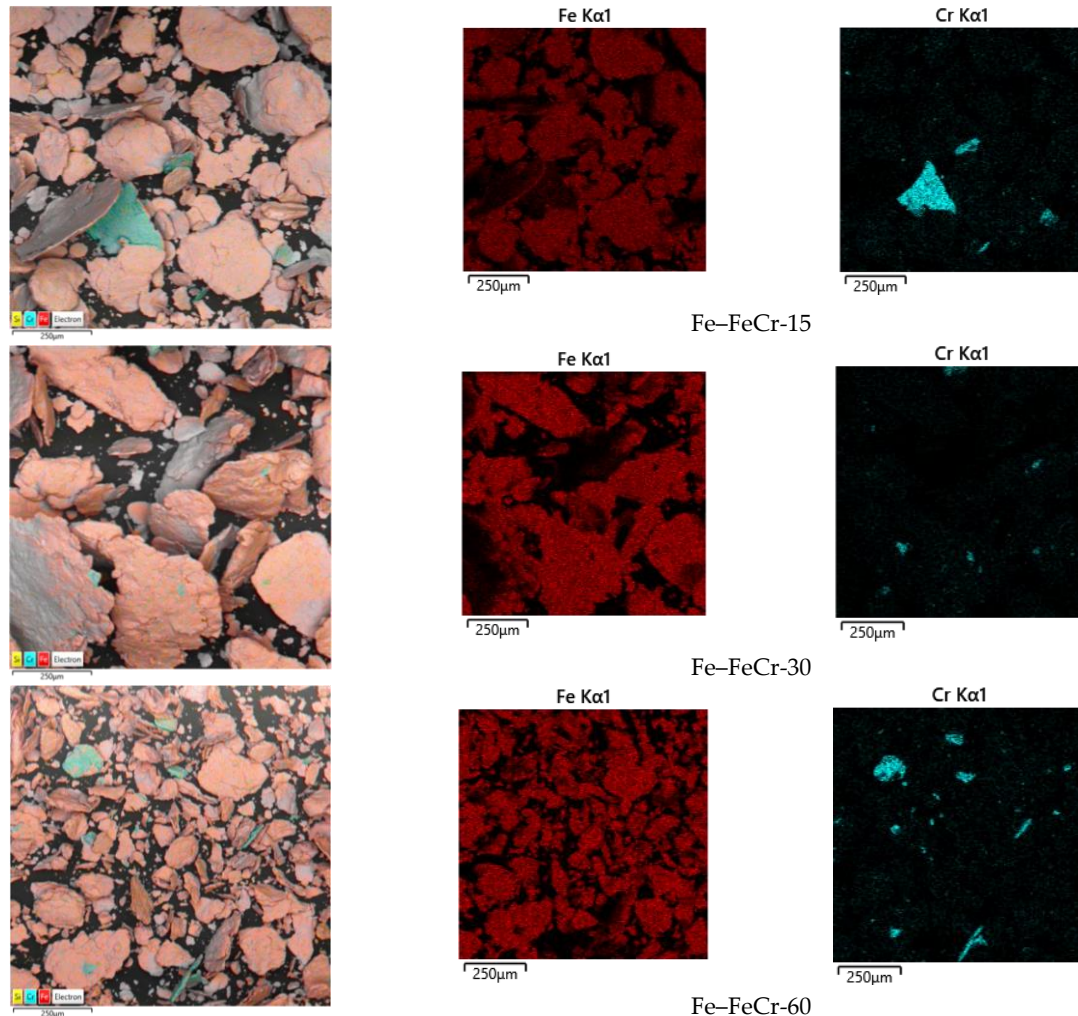


**Figure 2.** SEM micrographs and corresponding Fe and Cr maps of the Fe–FeCr-15, Fe–FeCr-30 and Fe–FeCr-60 precursor powders

*Note – prepared by the authors*

### Compactability and In Situ Nitride Formation

Compaction at pressures up to 700 MPa did not provide sufficiently high densification of the mechanically processed Fe–FeCr powders. The highest green densities were obtained at 700 MPa; therefore, only the results measured at this pressure are presented (Figure 3).

The relative green density was calculated as the ratio of the experimentally determined density to the theoretical density of the material, taken as 7.8 g/cm<sup>3</sup>, and expressed as a percentage. For comparison, the relative density of the compact produced from the initial pure Fe powder was approximately 91%. The data point at 0 min in Figure 3 corresponds to this initial pure Fe powder, which was pressed under the same conditions (700 MPa). The addition of LC FeCr and subsequent mechanical processing progressively reduced compactability, with relative density decreasing as the milling time increased.

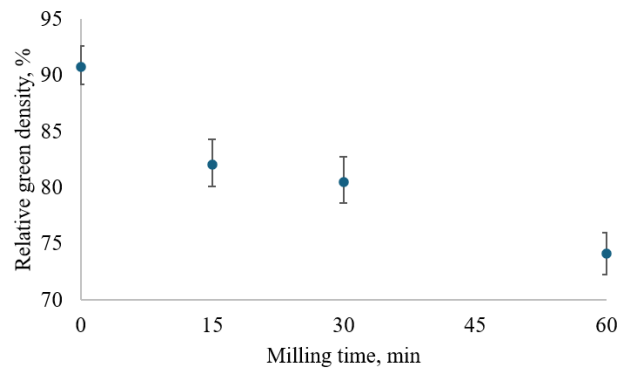


**Figure 3.** Green density of compacts prepared from the Fe–FeCr-15, Fe–FeCr-30, and Fe–FeCr-60 powders. The data point at 0 min corresponds to the initial pure Fe powder pressed under the same conditions (700 MPa). Values are means of three measurements ( $n = 3$ ); error bars represent standard deviations

The reduction in green density resulted from the combined effects of work hardening and particle-shape evolution. At the early stage, ductile Fe formed extended contact areas and closed pores effectively under pressure. Continued PCM processing increased dislocation density and residual strain, limiting further plastic deformation. Dispersed LC FeCr also increased the number of rigid Fe–FeCr and FeCr–FeCr contacts at which local stresses were concentrated. The activation that improved precursor dispersion and potential nitriding reactivity therefore reduced powder formability.

After nitrogen sintering, the Fe–FeCr-60 compact consisted of a continuous Fe-rich matrix containing discrete Cr-rich regions (Figure 4). Nitrogen overlapped primarily with the Cr-rich regions, demonstrating localized in situ nitriding of the mechanically activated LC FeCr precursor, whereas oxygen was concentrated at particle boundaries. Sintering produced dimensional shrinkage and partially healed preexisting cracks through interparticle neck formation, but it did not fully homogenize the material.

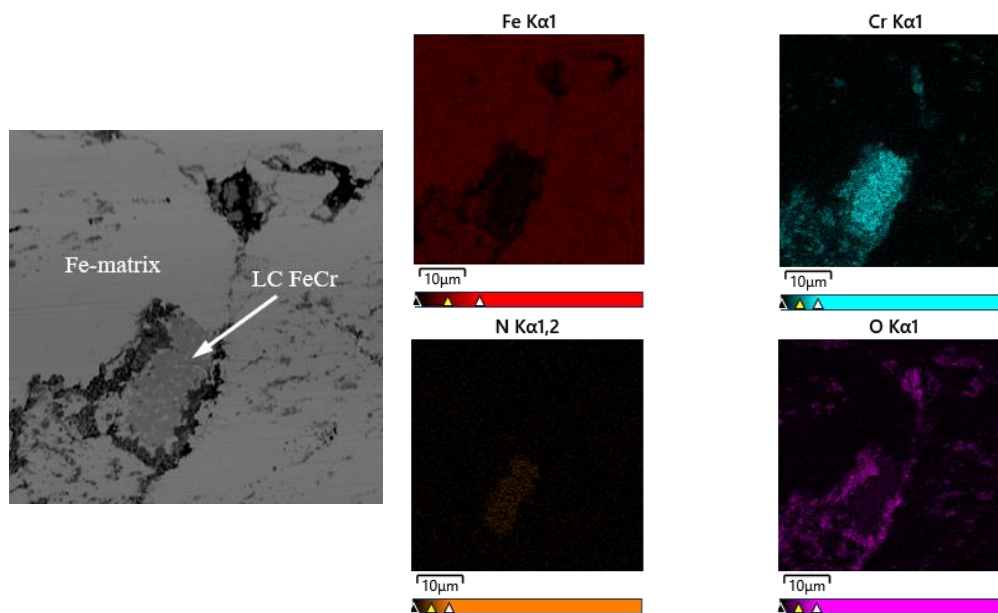


**Figure 4.** SEM micrograph and Fe, Cr, O, and N maps of the nitrogen-sintered Fe–FeCr-60 compact; arrows mark the point-EDS locations

**Table 3.** Local compositions of the nitrogen-sintered Fe–FeCr-60 compact, wt.%

| Spectrum  | Fe    | Cr    | N     |
|-----------|-------|-------|-------|
| Fe-matrix | 99.01 | 0.99  | —     |
| LC FeCr   | 7.34  | 82.64 | 10.02 |

The Fe-matrix spectrum contained 99.01 wt.% Fe and only 0.99 wt.% Cr, with no detectable nitrogen. In contrast, the LC FeCr-derived region contained 82.64 wt.% Cr, 7.34 wt.% Fe, and 10.02 wt.% N, confirming the preferential association of nitrogen with the Cr-rich constituent. The calculated atomic ratio of (Cr + Fe)/N was approximately 2.40. Considering the limited accuracy of EDS for light-element quantification and the possible contribution of the surrounding Fe-rich matrix to the analyzed volume, this composition is reasonably consistent with an  $M_2N$ -type Cr-rich nitride, provisionally assigned, on the basis of EDS compositional evidence only, as  $(Cr,Fe)_2N$ . The measured composition, corresponding to approximately 65.2 at.% Cr, 5.4 at.% Fe, and 29.4 at.% N, is also close to the metal-to-nitrogen stoichiometry expected for an  $M_2N$ -type nitride. Similar Cr-rich particles have previously been attributed to  $Cr_2N$  on the basis of Cr–N enrichment and thermodynamic considerations. Thermodynamic calculations further support chromium nitride formation: for the reaction  $2Cr + 0.5N_2 = Cr_2N$ , the calculated Gibbs free-energy change at 1150 °C was  $-405.86 \text{ kJ mol}^{-1}$ , indicating a strong thermodynamic driving force for  $Cr_2N$  formation under the considered conditions. Thus, the EDS composition together with the thermodynamic tendency toward  $Cr_2N$  formation supports (Yamasaki et al., 2018; Zhang et al., 2020), but does not crystallographically confirm, the proposed  $(Cr,Fe)_2N$  assignment.

The formation of the Cr–N-rich region indicates that in situ nitriding occurred during sintering in molecular  $N_2$ . Mechanical activation refined the LC FeCr particles, increased the Fe/LC FeCr interfacial area, and introduced deformation-induced defects that could facilitate nitrogen uptake and diffusion. Residual porosity also provided pathways for N transport toward the Cr-rich particles. However, the oxide film surrounding the LC FeCr-derived particle may have locally restricted direct metallic contact and hindered interfacial diffusion and bonding during sintering. Thus, LC FeCr acted simultaneously as the Cr source and as a dispersed precursor for in situ nitride formation. The duration of planetary centrifugal milling should therefore be selected by balancing enhanced nitriding reactivity against the deterioration of powder compactability and the possible influence of surface oxidation.

## CONCLUSIONS

Three Fe–FeCr powder states were produced by mechanical processing in a planetary centrifugal mill for 15, 30, and 60 min. Deformation and cold welding dominated the Fe–FeCr-15 and Fe–FeCr-30 conditions, whereas fracture became the principal mechanism in the Fe–FeCr-60 condition as work hardening progressed.

From 15 to 60 min, D50 decreased from approximately 90 to 53  $\mu\text{m}$ . The temporary increase in D90 at 30 min reflected the formation of coarse secondary agglomerates. EDS mapping of the Fe–FeCr-15 and Fe–FeCr-60 conditions demonstrated fragmentation and redistribution of Cr-rich LC FeCr without complete compositional homogenization.

Nitrogen sintering of the Fe–FeCr-60 compact at 1150 °C generated Cr–N-rich particles in situ. Their local composition was consistent with an  $M_2N$ -type Cr-rich nitride, provisionally assigned as  $(Cr,Fe)_2N$  on the basis of EDS compositional evidence only, while Fe–Cr–O shells remained at interfaces. Phase-sensitive characterization of the sintered material is still needed to confirm the nitride structure. Further process optimization should limit oxidation and identify a PCM duration that combines sufficient precursor activation with acceptable powder compactability.

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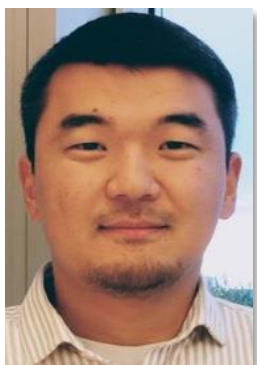
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