RASĀYAN *J. Chem.*
Vol. 19 | No. 2 | 1105-1109| April-June | 2026
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

INVESTIGATION OF STRUCTURAL, THERMAL, AND MECHANICAL PROPERTIES OF IRON-CROMIUM NANOSTRUCTURES

P. Endla✉
Department of Physics, SR University, Warangal-506371, (Telangana) India
✉Corresponding author: psm45456@gmail.com

ABSTRACT

This paper examines the structural development of iron -chromium alloy powder that was generated through the ball milling process. The primary emphasis is on the investigation of the lattice strain and nanocrystalline size in the synthesis of iron-chromium alloys by long milling. XRD analysis established that a body-centred cubic structure had been formed with the reflections mainly formed by the (110), (200) and (211) planes. The crystallite size, lattice strain and density of dislocation were determined by Williamson-Hall and modified Scherrer. It was found that the crystallite size steadily decreased with milling time, with the largest crystal size of approximately eleven nanometers after sixty hours and a corresponding increase in lattice strain, thus signifying increased internal stress and defect development. TEM images favoured the existence of pseudo-spherical nanograins in the same size scale. Other parameters obtained in the study by use of diffraction data include the Debye-Waller factor (DWF), the mean square amplitude of vibration, and Debye temperature (DT), and it showed a progressive loss of thermal stability as strain increases. The lowered DT was an indication of lattice softening due to long-lasting mechanical deformation. In general, the discussion proved that mechanical alloying is a successful method of refining particle size (PS), improving lattice strain (LS), and changing thermal and structural properties of the iron-chromium nanocrystalline alloy.

Keywords: Iron–Chromium Alloy, Mechanical Alloying, Lattice Strain, Nanocrystalline Size, X-Ray Diffraction, Transmission Electron Microscopy, Debye Temperature.

RASĀYAN*J. Chem.*, Vol. 19, No.2, 2026

INTRODUCTION

Oxide Dispersion Strengthened (ODS) systems, and other recent advancements in nanostructured Fe-Cr alloys, have shown excellent mechanical properties and radiation resistance and are of significant interest in terms of advanced nuclear and aerospace elements.[1,3] The possible use of MA in the synthesis of such ODS alloys has been validated by several studies that point to better oxidation resistance, high dislocation density and nanoscale uniformity.[4,6]The concept of estimating crystallite size from diffraction broadening was first introduced by Scherrer (1918), who proposed an equation correlating crystallite dimensions with X-ray diffraction (XRD) line broadening. According to Dinnebier and Billinge[7], a *crystallite* is a coherent diffracting region within a polycrystalline sample. The separation of strain and size contributions to peak broadening was later refined through the approach proposed by Hall, which was further developed by Mittemeijer and Scardi.[8] In diffraction analysis, the Gaussian and Lorentzian (Cauchy) functions are most frequently employed to describe strain and size broadening, respectively. However, running parallel and hence in most cases, making quantitative interpretation a difficult undertaking. The same study found the intermetallic phase in Fe–Cr alloys through XRD patterns by Untoro *et al.*[9], and estimated the crystallite sizes by the Scherrer equation without considering the lattice strain. To obtain more accurate characterisation, the Williamson Hall (W H) technique gives a more accurate model where the broadening of the peaks is decoupled between strain and size. Recent developments of this technique have also improved its precision to a point where the microstrain, the dislocation density, and the size of crystallites can be determined simultaneously.[10] Advanced engineering applications[11-15] require the mechanical and thermal properties of metallic alloys, which are important in measuring the distortions in the lattice due to stress during the milling of the crystalline material that can affect the XRD peak profiles. Inagaki *et al.*[16,17] was able to show that mechanical stresses of milled powder can act to influence the Debye-Waller

http://doi.org/10.31788/RJC.2026.1929591

Factor (DWF), a measure of atomic displacement within crystal lattices. The relationship between lattice strain, DWF, apparent volume and Debye temperature in pure metals and alloys has been widely studied in literature by Sirdeshmukh *et al*.[18,19], Gopi Krishna *et al.* twenty, and other authors twenty-one to twenty-four. These observations lead to the knowledge of how structure-property correlation is at the nanoscale. In the present study, Fe_8Cr_{20} alloy was chosen because there was a limited number of X-ray data available regarding its Debye-Waller factor, Debye temperature, lattice strain, preferred orientation, and dislocation density. The current work intends to present a detailed analysis of these parameters, and this is one of the first systematic X-ray examinations of Fe_8Cr_{20} alloy powder synthesised by mechanical alloying.

EXPERIMENTAL

Material And Methods

Fe and Cr powders of high purity (99.9) were measured to obtain the Fe_8Cr_{20} composition and were stored in an argon-filled glove box to avoid oxidation. The process control agent consisted of stearic acid (5 wt.%) to inhibit cold welding and preserve the dispersion of the particles. The powders and PCA were put into hardened steel ball-milled stainless-steel vials (1050 mm) and milled in a PM400 planetary ball mill at 300 rpm, at a ratio of 20:1 of ball to powder. Two samples were made: FeCr 40h (milled 40 hours) and FeCr 60h (milled 60 hours), and morphology, homogeneity, and alloying kinetics were periodically monitored.

Detection Method

The phase and structural examination of the mechanically alloyed Fe–Cr powders involved XRD in a diffractometer utilising Cu Kα radiation (λ = 1.5406 Å). The instrument operated at 40 kV and 30 mA, and data were gathered over a 2θ range of 20° to 80° with a scanning step of 0.02°. The diffraction patterns were examined to determine phase formation, crystallite size, lattice parameter, and strain. The WH method was applied to the diffraction data to distinguish the effects of PS and LS on peak broadening. This method gives a better estimate of lattice distortions and internal stresses generated during the mechanical alloying process. Morphological and microstructural analyses were conducted using TEM, which provided high-resolution images that confirmed nanoscale features and supported the crystallite size obtained from XRD.

RESULTS AND DISCUSSION

XRD Analysis

The XRD measurements of Fe, Cr, and $Fe_{80}Cr_{20}$ samples were done at room temperature using a Bruker D8 Advance diffractometer with Cu-Kα radiation (λ = 1.54056 Å). Data were collected with a step size of 0.02° 2θ and a counting time of 20 seconds per step. The diffraction patterns showed sharp peaks that matched the crystal planes of the bcc structure. The most intense reflections appeared at (110), (200), and (211). Fe had peaks at 2θ = 44.49°, 50.71°, and 73.36°; Cr had peaks at 44.29°, 50.41°, and 73.16°; and $Fe_{80}Cr_{20}$ had peaks at 44.39°, 50.31°, and 73.26°. These results confirmed the formation of a bcc structure in the alloy. No extra peaks were found for $Fe_{80}Cr_{20}$, which indicates a well-mixed and highly crystalline structure of Fe and Cr atoms. The oriented grains from the (110), (200), and (211) planes were then used to calculate the PS, LS, and dislocation density of the powders, offering information about their structural features. The diffraction peak for milled Fe-Cr 40h and FeCr 60h is shown in Fig.-1. The XRD patterns of FeCr 40h and FeCr 60h display strong diffraction peaks linked to scattering from the (1 1 0), (2 0 0), and (2 1 1) planes. While they differ in peak width, the scattering angles for the FeCr 40h sample as-milled are nearly the same as those for the FeCr 60h sample.

Characterization

TEMAnalysis

It is important to note that the XRD profile crystallite size value matched the average crystallite size of Fe-Cr. Additionally, the line integral breadth analysis method was only helpful for examining grain size. Therefore, it is necessary to observe the shape of the crystalline Fe-Cr grains to confirm the effectiveness of this analysis. The plan-view TEM micrographs in Fig.-12(a) and 12(b) show the arrangement of crystalline grains in milled powders of FeCr 40h and FeCr 60h, respectively. The average crystallite sizes

for Fe-Cr 40h and Fe-Cr 60h were about 11.44 nm and 9.78 nm, respectively, with grain sizes of less than 12 nm for both materials. The results from the XRD profile analysis, which showed that the line integral breadth method was effective for estimating the crystallite size of crystalline FeCr, supported the belief that the assessment based on TEM observation was correct.

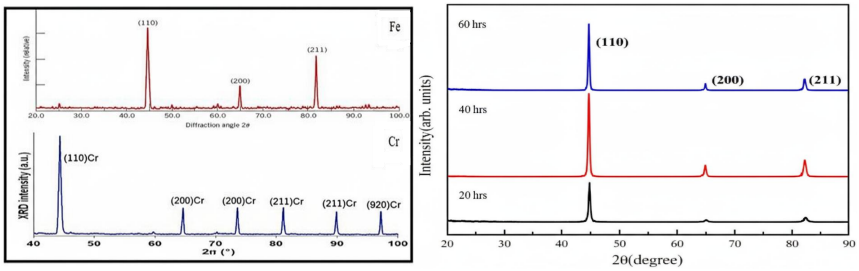

Fig.-1:XRDPattern forFeandCrPowder for 0, 20, 40 and 60 hours

According to the TEM micrograph, the powder is either pseudo-spherical or has irregular globular shapes as it is milled. The Fe and Cr powder experienced significant plastic deformation during the mechanical alloying process, altering the particle size, shape, and microstructure. For these initial powder mixtures, mechanical alloying starts with the flattening and work-hardening of chromium powder and the fragmentation of iron powder. After that, cold welding between powder particles occurs, leading to the formation of lamellar structures and the coarsening of the powder crystallite size distribution. The domain crystallite sizes revealed by TEM after the mechanical alloying process for FeCr 40h and Fe-Cr 60h were found to be in the range of 8.21-14.8 nm and 8.4-12 nm, respectively. The results indicated that with a milling period of 60 hours, the optimal powder crystallite size was determined to be 8.4 nm.

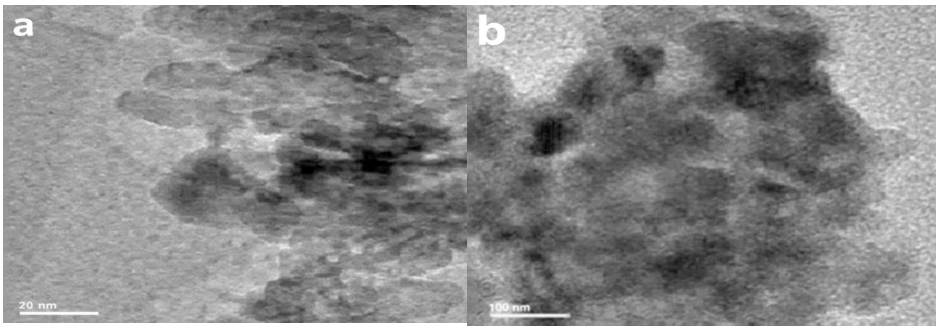

Fig.-2: TEMMicrographsfor(a)Fe-Cr40hand(b)FeCr60h

Thermal Properties

The mean particle size (D) of Fe, Cr and Fe80Cr20 is estimated by

$$D = \frac{0.89\ \lambda}{\beta \cos \theta} \quad (1)$$

The induced strain (ε) and dislocation density (δ) of the as-synthesised samples[21]:

$$\varepsilon = \frac{\beta \cos \theta}{4} \quad (2)$$

$$\delta = \frac{1}{D^2}$$

Modified Scherrer Method

$$\frac{\beta \cos \theta}{k\lambda} = \frac{1}{D} + \frac{4\varepsilon \sin \theta}{k\lambda} \quad (3)$$

Mechanical Properties

Using the DW theory, the directional Debye-Waller factor B is:

$$B = \frac{6h^2}{Mk_B D\theta_M} W(X)'' \quad (4)$$

The calculated values have been given in Table-1 and 2.

Table-1: Mechanical Properties of Fe, Cr and $Fe_{80}Cr_{20}$Powders

Material	Particle size(D nm)	$<u^2>(Å^2)$	$B(Å^2)$	$\theta_M(K)$	$(E_f)(eV)$
Fe	66.5	0.0042	0.35	424	4.02
Cr	43.8	0.0028	0.27	523	5.22
Fe_{80} Cr_{20}	35.6	0.0034	0.27	498	1.79

Table-2: Shows Calculated Values in the Present Investigation"

Material	T(hrs)	ε x 10^3	D (nm)	(δ) x 10^{15} (m^{-2})	t (nm)	$<u^2>(Å^2)$	$B(Å^2)$	θ_M (K)
$Fe_{80}Cr_{20}$	0	0.02	38.6	0.67	35.6	0.0034	0.27	498
	20	0.63	25.4	1.55	22.4	0.0035	0.28	486
	40	1.08	12.8	6.11	12.06	0.0037	0.29	478
	60	1.95	11.06	8.18	10.98	0.0039	0.31	469
	80	1.96	11.09	8.13	11.00	0.0040	0.32	462

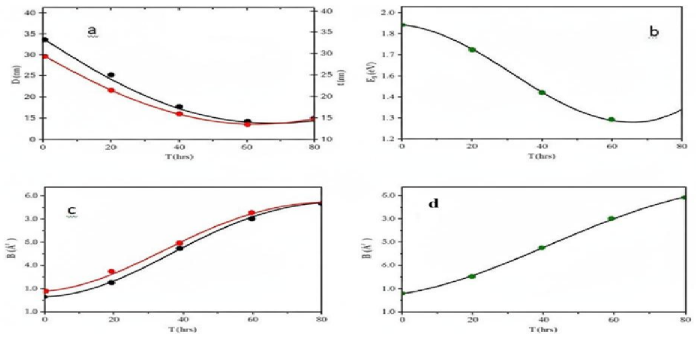

Fig.-4: Milling Time *Vs*(a) Particle Size, (b) Vacancy Formation Energy, (c) Debye –Waller Factor, (d) Mean Square Amplitudes of Vibrations, and (e) Debye Temperature for $Fe_{80}Cr_{20}$

CONCLUSION

To sum it up, the current research shows that mechanical alloying is a promising process to be used to produce nanocrystalline structures of iron-chromium alloys, which have a fine grain size, are stronger in lattice and have a better structural uniformity. Both XRD and TEM determined that there is the creation of nanoscale grains and augmentation of defect density, which adds to adjustable mechanical strength and thermal attributes. The highly rated and scalable path to create more advanced structural materials with superior performance is a strong feature of the process and is highly applicable in the field of industrial and technological implementation.

ACKNOWLEDGMENTS

The author, Dr. E. Purushotham, gratefully acknowledges SR University for providing research facilities and continuous support during this work.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9: Industry, Innovation, and Infrastructure*. The Author has contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

Purushotham Endla https://orcid.org/0000-0001-8412-5932

Cite this article as: P. Endla, *Rasayan J. Chem.*, 19(2), 1105-1109(2026), https://doi.org/10.31788/RJC.2026.1929591.

REFERENCES

1. H. Matsumoto, S. Hara, N. Nagata, and K. Ikeda, *Heterocycles*, **41**, 47(1995), https://doi.org/10.3987/COM-94-6871.
2. M. P. Phaniraj, D.-I. Kim, J.-H. Shim, and Y. W. Cho, *Acta Materialia*, **57**, 1856(2009), https://doi.org/10.1016/j.actamat.2008.11.063
3. A. Hirata, T. Fujita, Y. R. Wen, J. H. Schneibel, C. T. Liu, and M. W. Chen, *Nature Materials*, **10**, 922(2011), https://doi.org/10.1038/nmat3123
4. M. K. Miller, C. M. Parish, and Q. Li, *Materials Science and Technology*, **29**, 1174(2013), https://doi.org/10.1179/1743284713Y.0000000219
5. R. K. S. Raman, *Metals*, **11**, 695(2021), https://doi.org/10.3390/met11050695
6. D. Feriyanto and S. Zakaria, *International Journal of Advanced Technology in Mechanical, Mechatronics and Materials*, **1**, 1(2024).
7. R. E. Dinnebier and S. J. L. Billinge, Powder Diffraction: Theory and Practice, Royal Society of Chemistry Publishing, United Kingdom (2008), https://doi.org/10.1039/9781847558237
8. E. J. Mittemeijer and P. Scardi, Diffraction Analysis of the Microstructure of Materials, Springer, Berlin (2004), https://doi.org/10.1007/978-3-662-09291-0
9. P. Untoro, D. Sebayang, and H. Saryanto, In Proceedings of the 7th ASEAN Microscope Conference, BATAN, Indonesia (2009).
10. S. Dutta and P. K. Das, *Scripta Materialia*, **217**, 114768(2022), https://doi.org/10.1016/j.scriptamat.2022.114768
11. D. B. Sirdeshmukh, L. Sirdeshmukh, and K. G. Subhadra, *Bulletin of Materials Science*, **35**, 939(2012), https://doi.org/10.1007/s12034-012-0358-8
12. M. Inagaki and K. Kuroda, *Journal of Alloys and Compounds*, **856**, 157443(2021), https://doi.org/10.1016/j.jallcom.2020.157443
13. K. Tkáčová and P. Baláž, *Materials Chemistry and Physics*, **267**, 125098(2021), https://doi.org/10.1016/j.matchemphys.2021.125098
14. D. B. Sirdeshmukh and G. Krishna, *Indian Journal of Pure and Applied Physics*, **58**, 120 (2020).
15. P. Ezhil, Materials Today: Proceedings, 33, 294 (2021), https://doi.org/10.1016/j.matpr.2020.05.691
16. M. Inagaki and K. Nakano, *Powder Metallurgy*, **63**, 315(2020), https://doi.org/10.1080/00325899.2020.1776373
17. M. Inagaki, *Acta Crystallographica Section A: Foundations and Advances*, **78**, 345(2022), https://doi.org/10.1107/S2053273322003405
18. D. B. Sirdeshmukh and K. G. Subhadra, *Journal of Physics and Chemistry of Solids*, **142**, 109012(2020), https://doi.org/10.1016/j.jpcs.2020.109012
19. B. G. Krishna and M. P. Reddy, *Journal of Materials Science: Materials in Electronics*, **32**, 18074(2021), https://doi.org/10.1007/s10854-021-05973-2
20. B. G. Krishna and R. M. Rao, *Materials Characterization*, **179**, 111345(2021), https://doi.org/10.1016/j.matchar.2021.111345
21. C. V. Reddy and P. Kumar, *Materials Today Energy*, **20**, 100955(2021), https://doi.org/10.1016/j.mtener.2021.100955
22. N. Sharma, *Results in Physics*, **28**, 105069(2022), https://doi.org/10.1016/j.rinp.2021.105069
23. D. Kumar and R. Singh, *Journal of Alloys and Compounds*, **918**, 165787(2022), https://doi.org/10.1016/j.jallcom.2022.165787
24. A. Gupta, *Materials Chemistry and Physics*, **304**, 127986(2023), https://doi.org/10.1016/j.matchemphys.2023.127986
25. I. E. Dag, M. Kaya, and O. Yilmaz, *Advanced Engineering Materials*, **27**, 1258(2025), https://doi.org/10.1002/adem.202402583
26. J. Miller, A. Thomas, and L. Zhang, *Journal of Materials Research*, **40**, 123(2025), https://doi.org/10.1557/s43578-025-0123-5
27. M. B. Ali and S. Shukla, *Materials Today: Proceedings*, **72**, 114(2024), https://doi.org/10.1016/j.matpr.2024.01.114

[RJC-9591/2025]