



Research Article

# High-Resolution Neutron Diffraction Analysis of Residual Stresses in Oxide Dispersion Strengthened FeNiCrY<sub>2</sub>O<sub>3</sub> Cast Alloys for Advanced Nuclear Reactor Applications

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**Abstract:** High-temperature resistant materials have been developed, including Fe-base austenitic steel with high Ni-Cr content. These materials are modified into oxide dispersion strengthened (ODS) steel incorporated with a small amount of rare earth element (REE) oxides. ODS alloys are a mixture of a metal matrix with uniformly distributed small oxide particles. The alloys show great promise as structural materials for modern engineering applications in advanced- energy and nuclear reactors. A comprehensive understanding of individual mechanical properties and residual stress distribution remains elusive. Therefore, this research aimed to show the influence of Y<sub>2</sub>O<sub>3</sub> dispersion on the crystal structure and residual stress in FeNiCr Y<sub>2</sub>O<sub>3</sub> cast alloys with varying ODS compositions ranging from 0 to 2.0 wt.% Y<sub>2</sub>O<sub>3</sub>. Alloys were synthesized by powder metallurgy and melt casting followed by characterization using neutron diffraction as well as standard metallographic methods. During analysis, neutron was powerful for probing solid materials, among other things. The product penetrated deep inside, which made it possible to research bulk properties and internal structures of thick samples or materials encased in containers. Diffraction profile analysis showed that cast alloys signified 'matrix-cluster' composite crystal morphology and improved elemental mapping of the microstructure. Moreover, stress-strain curves moderately estimated optimum Y<sub>2</sub>O<sub>3</sub> content to be 1.0 wt.%. Y<sub>2</sub>O<sub>3</sub> dispersion induced lattice compression in the austenitic FeNiCr matrix, signifying tensile residual stress in the crystals. Distribution of residual stress in alloys was related to mechanical and

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performance properties. In conclusion, most recommended materials for further fabrication as FeNiCr Y<sub>2</sub>O<sub>3</sub> ODS cast alloys was 57Fe25Ni17Cr1.0Y<sub>2</sub>O<sub>3</sub> system.

**Keywords:** FeNiCrY<sub>2</sub>O<sub>3</sub> ODS casting; Microstructure; Neutron diffraction; Residual stress; Y<sub>2</sub>O<sub>3</sub>

## 1. Introduction

Materials suitable for high-temperature environments are advanced nuclear reactors (Was et al., 2019; Zinkle and Was, 2013), which mainly include ferritic and martensitic (F/M)-type superalloy steels. These materials show poor creep properties and have lower resistance to swelling at high temperatures compared to superalloy austenitic steel contrast. Superalloy austenitic steel has a lower hardness than ferritic and martensitic steel, signifying that its toughness still requires improvement. This behavior improves the addition of nano- or micro-powders of rare earth elements (REE), such as Yttria (Y<sub>2</sub>O<sub>3</sub>), leading to 'oxide dispersion strengthened' (ODS) steel alloys. The hardness of ODS materials increases due to nano-dispersion of Yttria grains, which helps prevent dislocation movement during creep deformation (Du et al., 2024; Xu et al., 2023; Kishorekumar et al., 2018).

The search for advanced materials that can withstand extreme conditions is challenging and has attracted much interest in designing as well as developing advanced nuclear reactors (Aziz et al., 2024; Gougar et al., 2020; Buckthorpe, 2017; Zinkle and Was, 2013). Defining temperature limits is critical for safety requirements, for normal or abnormal operations. This occurs because corrosion at high temperatures has a direct impact on the other mechanical parameters important for safety (Zhang et al., 2019). Numerous candidate materials are investigated for individual use in a high-temperature environment (Yusupandi et al., 2024; Arshad et al., 2022). Among the potential candidates, austenitic stainless steel (ASS) alloys, particularly 56Fe25Ni16.55Cr composition, which is later refined to 58Fe25Ni17Cr, show further improvement. This increment showed resistance to high-temperature corrosion up to 850°C at a rate of 0.06 mm/year (Mustofa et al., 2020; Dani et al., 2020; Parikin et al., 2019). Research to improve the performance of the model by incorporating a rare earth metal oxide, Y<sub>2</sub>O<sub>3</sub>, has been investigated (Parikin et al., 2021).

Some high-temperature resistant materials, such as 58Fe25Ni17Cr austenitic steel with high Ni-Cr content, have been recently developed (Dani et al., 2019). FeNiCr-based superalloy steels have further been developed to ODS alloys through melt-casting, instead of through powder metallurgy methods (Raman et al., 2016). Moreover, synthesis by a pre-linking method was introduced (Parikin et al., 2024), in which Fe+Y<sub>2</sub>O<sub>3</sub> variable-dependent composition was designed to be physically adjacent to cast alloys. Due to varying melting temperatures of main components of alloys (Fe, Ni, Cr, and Y<sub>2</sub>O<sub>3</sub>) the components develop separately into two distinct phases, i.e. fcc-austenite (FeNiCr) and cubic (Y<sub>2</sub>O<sub>3</sub>), forming composite materials with unique morphology. However, the presence of these oxide particles in the matrix introduces residual stress impacting reliability and performance of materials (Brewer et al., 2015; Ruan et al., 2012; Oka et al., 2007).

Neutron diffraction method is a powerful non-destructive method for characterizing residual stress in crystalline materials. An overview of the application of neutron diffraction methods in residual stress analysis has been described in (Parikin et al., 2009). Additionally, neutron has a high penetration depth in many materials, allowing research of bulk properties and internal structures of thick samples or materials encased in containers. Different from neutron, X-rays have much lower penetration depth, making the procedure more suited to surface analysis or thin samples. Following this discussion, residual stress is a major factor influencing microstructure and mechanical properties that can be beneficial or detrimental depending on sign, magnitude, as well as loading conditions (Zang et al., 2024; Andoko et al., 2024; Naserinejad et al., 2023; Guo et al., 2020; Li et al., 2019). Under tensile conditions, residual tensile stress on surfaces poses a risk, potentially causing fatigue-induced crack growth (Dong et al., 2023; Kühne et al., 2021; Statti et al., 2021). To address this issue, diffraction methods, particularly neutron diffraction, can be used to examine lattice of strains (Em et al., 2024; Yan et al., 2022; Mo et al., 2018; Tanaka et al., 2002).

This research aims to obtain ODS cast alloy, specifically FeNiCrY<sub>2</sub>O<sub>3</sub> as superior materials for high-temperature applications. The objective of the analysis is to measure lattice crystals and analyze residual stress in ODS alloys using neutron diffraction and Rietveld analyses. By systematically investigating different oxide contents in FeNiCrY<sub>2</sub>O<sub>3</sub> ODS cast alloys, correlations between residual stress and microstructure properties with mechanical properties can be developed.

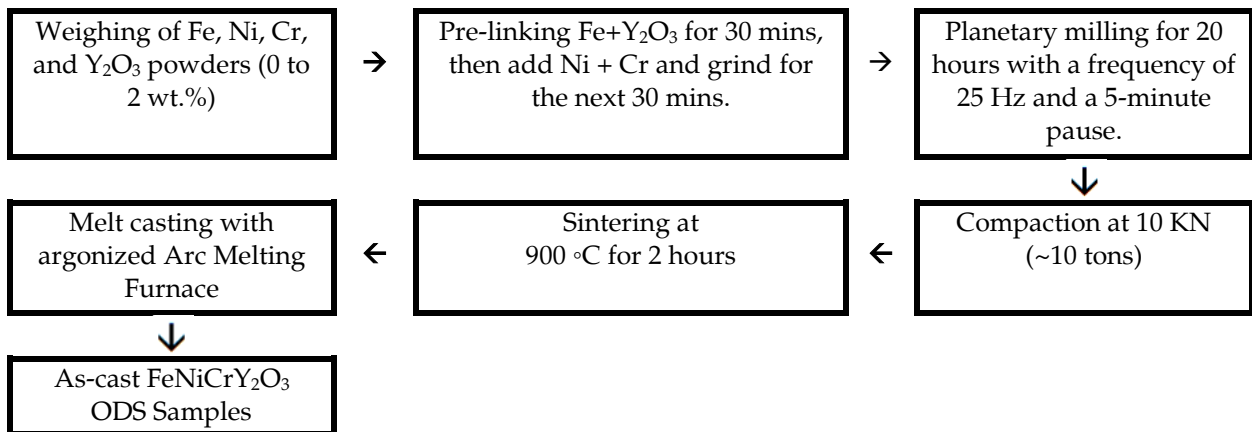
## 2. Methods

### 2.1. Used materials for experiments

The initial precursors used to synthesize FeNiCrY<sub>2</sub>O<sub>3</sub> ODS cast alloys were shown in Table 1. Pure Fe and Cr powders were supplied by Alfa Aesar with purities of 98 and >99, 99.8, and 99.99%. Following the analysis of this research, each precursor had a varying property. The manufacture of samples of ODS cast alloys began with weighing the high-purity powder, using a precision balance. During the process, a one-hour pre-linking method and a twenty-hour planetary ball mill were applied to mix a minimal amount of Y<sub>2</sub>O<sub>3</sub> oxide for uniform dispersion in cast alloys with a homogeneous particle size Figure 1. Subsequently, the synthesis was conducted according to the scheme presented in the Figure and described chronologically hereafter.

**Table 1** Composition of the initial high-purity precursors used to synthesize ODS FeNiCrY<sub>2</sub>O<sub>3</sub> alloy

Powder	Purity	Crystal Structure (at 25°C)	Particle Size (µm)	Density (g.cm <sup>-3</sup> )	Melting point (°C)	Source
Fe	98%	<i>bcc</i>	44.00	7.80	1535	Alfa Aesar, US
Cr	>99%	<i>bcc</i>	44.00	7.14	1435	Alfa Aesar, US
Ni	99.8%	<i>fcc</i>	45.00	8.90	1907	Sigma-Aldrich, US
Y <sub>2</sub> O <sub>3</sub>	99.99%	<i>cubic</i>	0.05	5.01	2410	Sigma-Aldrich, US



**Figure 1** Schematic showing preparation of FeNiCrY<sub>2</sub>O<sub>3</sub> cast alloys with ODS compositions varying from 0 to 2.0% by weight of Y<sub>2</sub>O<sub>3</sub>

### 2.2. Mechanical alloying

The composition of added Y<sub>2</sub>O<sub>3</sub> powder was adjusted to match the reduction in composition of Fe powder. High-grade metal powders of Fe, Cr, Ni, and Y<sub>2</sub>O<sub>3</sub>, as shown in Table 2, were used in casting process. In addition, 25-gram precursor was prepared using Fe+Y<sub>2</sub>O<sub>3</sub> pre-linking route, which included crushing and grinding in a mortar for 30 minutes manually.

**Table 2** Chemical composition of synthesized ODS FeNiCrY<sub>2</sub>O<sub>3</sub> specimens. The specimens were obtained by vacuum electric arc furnace (EAF) of mechanically alloyed powders

Specimen	Composition (wt.%)			
	Fe	Ni	Cr	Y <sub>2</sub> O <sub>3</sub>
FeNiCr	58.0	25.0	17.0	0.0
FeNiCr-03Y <sub>2</sub> O <sub>3</sub>	57.7	25.0	17.0	0.3
FeNiCr-05Y <sub>2</sub> O <sub>3</sub>	57.5	25.0	17.0	0.5
FeNiCr-07Y <sub>2</sub> O <sub>3</sub>	57.3	25.0	17.0	0.7
FeNiCr-10Y <sub>2</sub> O <sub>3</sub>	57.0	25.0	17.0	1.0
FeNiCr-15Y <sub>2</sub> O <sub>3</sub>	56.5	25.0	17.0	1.5
FeNiCr-20Y <sub>2</sub> O <sub>3</sub>	56.0	25.0	17.0	2.0

Since Fe was the dominant element in system of alloys, Y<sub>2</sub>O<sub>3</sub> grains were close to Fe grains (Parikin et al., 2024). Subsequently, Ni and Cr powders were continuously added as well as subjected to crushing grinding for an additional 30 minutes. Finally, mechanical grinding for 20 hours in Planetary Mill with ball-to-powder ratio of 12:1 and frequency of 25 Hz—5 mins pause was applied.

### 2.3. Sintering and Smelting

The milled powder was compacted into pellets, each with diameter of 10 mm and thickness of 5 mm, using hydraulic pressurizer (Instron 5569) with pressure of 100 kN which was equivalent to 10 tons load. These pellets were subsequently sintered in vacuum furnace (Carbolite Gero HTRV 16/75/550) at temperature of 900 °C for 2 hours. This sintering process was performed to consolidate the powder and improve the bonding between metal matrix as well as oxide particles. Following sintering, pellets were smelted in an EAF operating at current of 200 A and voltage of 18 V, with an argon flow of 3.5 bars. Ingot specimen was flipped four times during smelting process to ensure uniform heating and melting.

### 2.4. Characterization Methods

#### 2.4.1. Microstructure observation

Microstructure of the specimens was examined using a variety of microscopy methods. These processes included Olympus BX51DP26 Light Optical Microscope (LOM), Jeol SEM 2650LA Scanning Electron Microscope (SEM) equipped with Energy-Dispersive X-ray Spectroscopy (EDX) detector, and Hitachi HT7700 Transmission Electron Microscope (TEM). The specimens during the analysis were prepared following standard metallography procedure. Initially, the specimens experienced surface grinding using abrasive papers of different grit sizes, ranging from coarse (80) to fine grain (5,000). This procedure was followed by polishing process conducted on pad covered with diamond powder paste with particle size of 1.0 μm. When the surfaces were smooth, the specimens were carefully cleaned using alcohol. The specimens were then etched using an aqua-regia solution for 1-2 minutes to identify individual microstructures. In addition to these procedures, Hitachi FIB 2200 Focused Ion Beam (FIB) was used for the preparation of TEM specimens. This comprehensive method ensured a detailed and accurate observation of microstructure of specimens.

#### 2.4.2. High-resolution neutron powder diffraction

Crystal structure of specimens of FeNiCrY<sub>2</sub>O<sub>3</sub> ODS cast alloys was examined using Sumitomo Rigaku high-resolution neutron powder diffractometer (HRPD) at G.A, and Siwabessy Research Reactor (RSG-GAS) which was operated at power of 15 MW. The solid weight of each deployed melt-cast ODS specimen ranged from 6 to 8 grams. Additionally, NPD (new products development) data were collected using a wavelength of 1.82 Å with 2θ ranging from 2.5° to 162.5°.

### 2.4.3. Rietveld analysis

Rietveld analysis commonly called Rietveld refinement was a method in crystallography that was generally used to smoothen crystal structure models by matching all powder neutron and X-ray diffraction patterns through the powder. This method was widely used in determining crystal structure of materials and was capable of providing information i.e., atomic positions, thermal vibrations, diffraction planes, crystallite size, and lattice spacings.

NPD data were analyzed using Rietveld method to obtain lattice and anisotropic mean-square displacement (U) parameters for each phase as well as specimen. Average strain ( $\epsilon_{av}$ ) and average stress ( $\sigma_{av}$ ) were calculated from these parameters. During the process, structural data analysis was conducted using a Rietveld structure refinement program, and Materials Analysis Using Diffraction (MAUD). This MAUD program analyzed diffraction spectra and obtained the structure of crystal, quantity, phase microstructure, texture, as well as residual stress of materials (Lutterotti, 2000). The initial lattice and U parameters to conduct Rietveld analysis for each specimen were shown in Table 3.

## 3. Results and Discussion

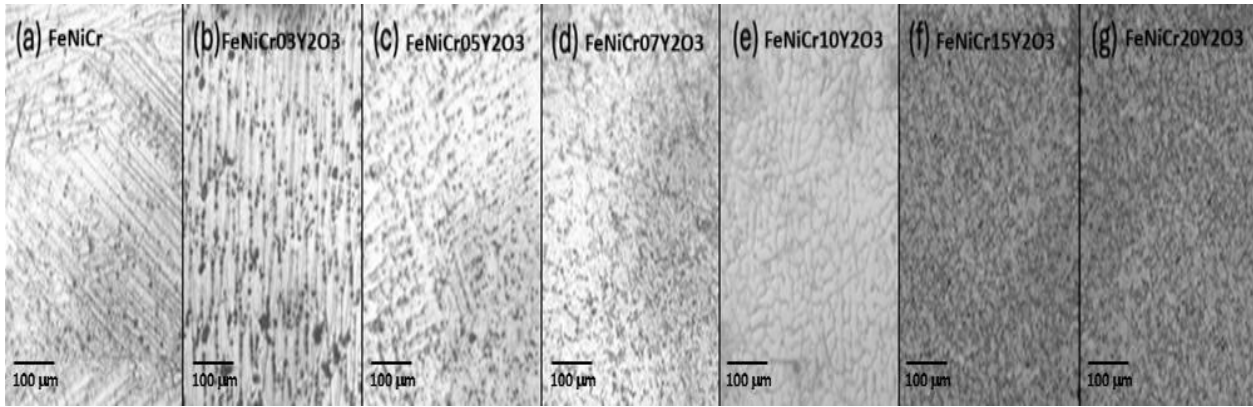
### 3.1. Microstructure Observations

#### 3.1.1. Optical microscopy

Figure 2 showed the effect of  $Y_2O_3$  dispersion on microstructure of melt-casted FeNiCr $Y_2O_3$  ODS alloys. FeNiCr cast alloys without  $Y_2O_3$  dispersion, as shown in (a), signified needle-like grains that were typical for ASS. The grains were also visible in FeNiCr alloys with 0.3 and 0.5 wt.%  $Y_2O_3$  dispersion. However, with the increase of  $Y_2O_3$  content, the needle-like grains were transformed into circular dendritic formations with wider grain boundaries, as shown in the specimens with 0.7 (d) and 1.0 wt.% (e)  $Y_2O_3$ . Although circular dendritic formations were also expected from the specimens with 1.5 (f) and 2.0 wt.% (g)  $Y_2O_3$ , coarse grains with different orientations were observed. The surface microstructure showed the formation of dispersoids in a micrometer-sized matrix. The dispersoids had diameter range of about 40–80  $\mu m$  that were present along the grain boundaries. This process showed the existence of discontinuity in matrix material, generating internal stress.

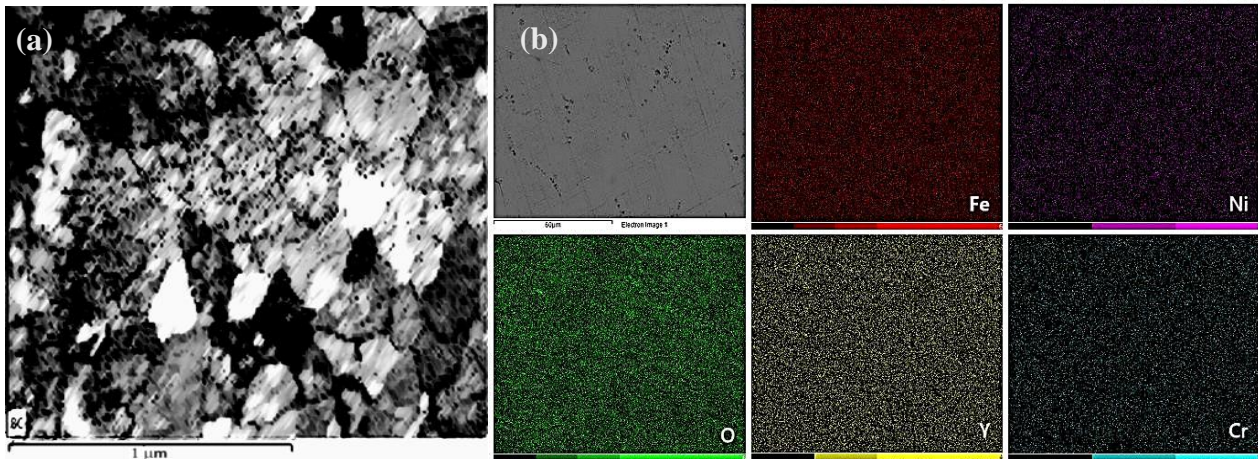
**Table 3** Initial parameters for the structural refinements of the ODS FeNiCr $Y_2O_3$  cast alloy

Crystallographic data	Phase-1	Phase-2
Formula	FeNiCr	$Y_2O_3$
Space group	Fm-3m (I-225)	Ia-3 (I-206)
Lattice parameter:		
a ( $\text{\AA}$ )	3.599724	10.599373
b ( $\text{\AA}$ )	3.599724	10.599373
c ( $\text{\AA}$ )	3.599724	10.599373
$\alpha, \beta, \gamma$ ( $^\circ$ )	90, 90, 90	90, 90, 90
Cell volume ( $\text{\AA}^3$ )	46.645262	1,190.804663
Atomic number in unit cell, Z	4	144
Atomic position, x, y, z	Fe1: 0.00, 0.50, 0.50	Y1: 0.26, 0.06, 0.21 Y2: 0.25, 0.00, 0.53 O1: 0.16, 0.33, 0.13
$U_o$ parameter	0.0011	0.19701
Young modulus (GPa) (Moreau, et al., 2008)	198	63.5



**Figure 2** Effect of  $Y_2O_3$  dispersion on microstructure of melt-casted ODS  $FeNiCrY_2O_3$  cast alloys. Optical microscope image with 100x magnification of the inner part of alloys with different dispersion of  $Y_2O_3$ : (a) 0.0 wt.%, (b) 0.3 wt.%, (c) 0.5 wt.%, (d) 0.7 wt.%, (e) 1.0 wt.%, (f) 1.5 wt.%, and (g) 2.0 wt.%

Figure 3 showed grain fineness achieved through cast fusion process under austenitic  $FeNiCr$  melting conditions (approximately  $1450\text{ }^\circ\text{C}$ ). This method effectively and compactly bonded  $Y_2O_3$  oxide in cast ODS alloy, which contrasted sharply with ODS produced by conventional powder metallurgy methods. The metallurgy method included cold sintering (CS), metal sintering (MS), extrusion (XTR), hot pressing (HP), hot isostatic pressing (HIP), and spark plasma sintering (SPS). (Kishorekumar et al., 2018). In addition, the image showed that alloys without  $Y_2O_3$  had needle-like grains, signifying a high degree of directional compaction. As dispersion of  $Y_2O_3$  increased, the needle-like grains were transformed into cycle-like dendritic formations with wider grain boundaries, which showed more isotropic compaction and higher resistance to grain growth.

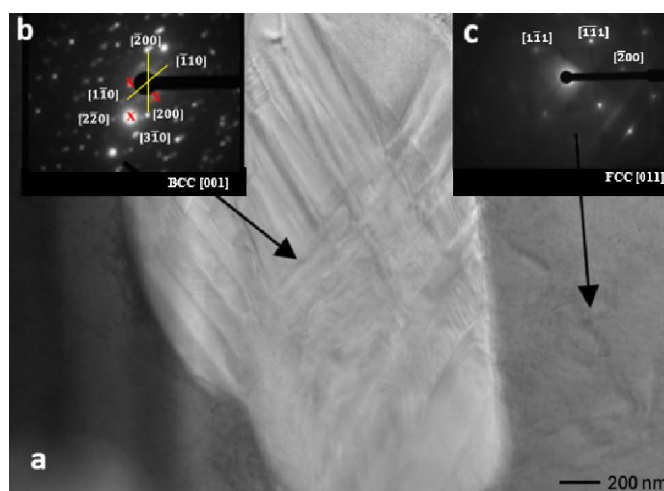


**Figure 3** (a) SEM image showing the structure of  $-FeNiCr$  matrix and yttria clusters, featuring an average globular austenite grain size of approximately  $\sim 1.1$  microns, with dispersion of nano oxides both in the grains and along grain boundaries. (b) EDS mapping observation of the inner part of melt-casted ODS  $FeNiCr10Y_2O_3$  alloys

The image showed the uniform dispersion of Y elements, which were strongly associated with Fe. This signified the effectiveness of pre-linking method for achieving homogeneous distribution of  $Y_2O_3$  in alloys. Elemental analysis enumerated that, Fe, Ni as well as Cr had about 23.21, 57.09, and 16.55 wt.%, respectively. In addition, Y (yttrium) about 1.32 wt.%, and O (oxygen) having approximately 1.83 wt.% contributed to system of alloys, signifying the content of  $Y_2O_3$ .

### 3.1.2. SEM/EDS observation

Based on SEM observation of specimens of FeNiCr alloys with different ODS compositions (0 – 2.0 wt.%  $Y_2O_3$ ), the analysis found that the specimens showed similar morphology as implied by LOM observation. In addition to the process, EDS mapping of these specimens signified interesting results related to the correlation of Fe and Y dispersion. Figure 3 showed SEM/EDS mapping of melt-casted FeNiCr $Y_2O_3$  ODS alloys with 1.0 wt.%  $Y_2O_3$  dispersion. Similar EDS mapping results were observed from all specimens with  $Y_2O_3$  content. Moreover, element mapping of Y looks closely related to Fe, which signified the effectiveness of pre-linking method for achieving a homogeneous distribution of  $Y_2O_3$  in alloys.



**Figure 4** (a) TEM image of FeNiCr10 $Y_2O_3$  alloys showing structural identification of matrix and dispersoid in a few nanometers, (b) and (c) showed indexed diffraction pattern for a BCC [001] and FCC [011] crystals (Globalsino, n.d.). The spacing ratio of the main points was shown as well as the angle between normal planes of the main planes,  $45^\circ$  for zone [001] and  $54.74^\circ$  [011], respectively. Moreover, the forbidden reflections (b) in zone [001] were shown by the red-cross outside  $45^\circ$  angle path. The outcome was due to cubic  $Y_2O_3$  bixbyite structure with C2 and C3i/S6 symmetry sites (Jadhav et al., 2017; Gaboriaud et al., 2015)

### 3.1.3. TEM observation

FeNiCr $Y_2O_3$  cast alloys were developed by melt casting methods in an argon atmosphere. Minimal amounts of yttria nano-dispersion were incorporated into alloys, forming a 'matrix-cluster' crystal composite cast alloy. In addition, the 'matrix-cluster' arrangements were FeNiCr austenite (FCC) and  $Y_2O_3$  oxide (cubic), including crystalline phases that grew in alloy. The morphology and mapping of alloys showed a stable ODS structure with 'globular' yttria dispersed fragmentarily in it. During the process, individual melting temperatures and densities were significantly different. The yttria was not compounded to form fused materials, as it still stood alone as clusters in a complex cubic phase. Figure 4 showed TEM image of inter-alloying between  $Y_2O_3$  and austenitic matrix in FeNiCr10 $Y_2O_3$  ODS cast alloys. A similar microstructure was expected for the other specimen of ODS alloys with different  $Y_2O_3$  contents. TEM image showed a coarsening of cubic  $Y_2O_3$  particles in the center of the austenitic matrix. Moreover, the selected region diffraction patterns of austenitic matrix and cubic  $Y_2O_3$  particles were obtained along [011] as well as [001] crystallographic directions. These two phases formed the composite structure of ODS cast alloy. The discontinuity in crystal structures caused a lattice stretching dynamic in materials.

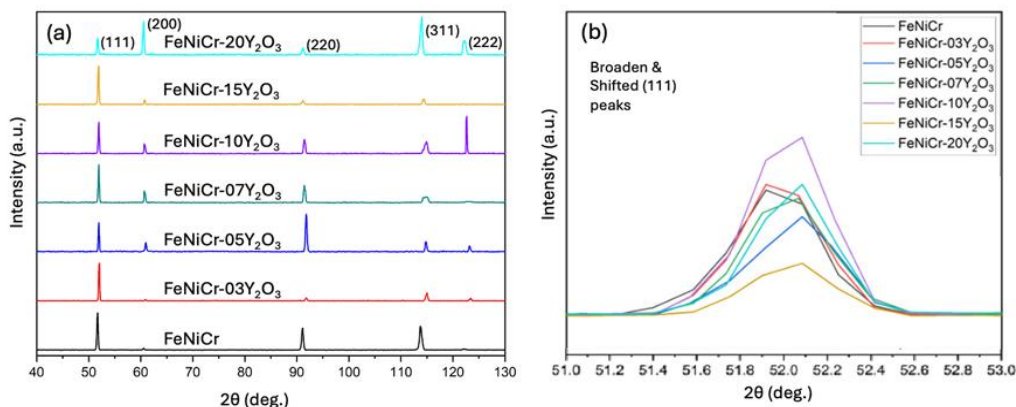
TEM in Figure 4(a) showed that the size of  $Y_2O_3$  dispersoid was in the micrometer range, as shown by brighter region in the image. Diffraction pattern obtained from  $Y_2O_3$  region signified BCC [001] zone axis pattern based on the database, shown by  $45^\circ$  angle formed by diffraction vectors  $[200]$  and  $[\bar{1}10]$  in Figure 4(b) (Globalsino, n.d.). Moreover, forbidden reflections were observed,

marked by red cross in Figure 4(b), from cubic  $Y_2O_3$  bixbyite structure with C2 and C3i/S6 symmetry sites. (Jadhav et al., 2017; Gaboriaud et al., 2015). Diffraction pattern from matrix region in Figure 4(c) showed FCC [011] structure as identified in the database. This outcome was signified by  $54.74^\circ$  angle formed through diffraction vectors  $[200]$  and  $[\bar{1}\bar{1}1]$ . This outcome was expected from FeCrNi matrix which commonly had FCC crystal structure (Fussik et al., 2018; Williams and Carter, 2009). Figure 4(a) showed that alloys had a combined austenite-FeNiCr (fcc) structure along [011] crystallographic direction, while cubic-ytria crystals were oriented near [001], as signified by the black bars blocking direct beam. These two phases had different thermal properties and interacted with each other, causing the rise of internal stress in ODS casting alloy. Panels in 4(b)-(c) showed the indexing of selected Bragg reflection patterns obtained from cubic lattice in 4(a). The simple reciprocal chamber of cubic shown in 4(b) had a simple cubic diffraction pattern. Therefore, there were some forbidden reflections in [001] axis zone, arising from complex multi-atomic unit cell structures (Jin and Kim, 2021; Gaboriaud et al., 2015). Following the discussion, the hkl-reflections in during this analysis took any integer. In Figure 4(c), the pattern at zone axis [011] matched FCC pattern. During the research, indexing spots explained how to identify the selected Bragg reflection patterns to determine Miller indices crystal planes (Williams and Carter, 2009). Cubic lattice shown in 4(c), raised face centered diffraction patterns, allowing Bragg reflection to have  $h+k+l=2n$ . The difference between indexing of Bragg peaks in 4(b) and 4(c) was due to lattice types, which were I as well as P for panels in 4(b) and 4(c) respectively (Olaoye, 2015).

### 3.2. Rietveld Analysis

#### 3.2.1. FeNiCr $Y_2O_3$ ODS cast alloys profiles

Figure 5 (a) showed neutron diffraction patterns of FeNiCr $Y_2O_3$  ODS cast alloys with the  $2\theta$  ranging from  $40^\circ$  to  $130^\circ$ . FeNiCr alloys signified a face-centered cubic (fcc) crystal system reviewed by five main diffraction peaks. Additionally, the corresponding planes were (111), (200), (220), (311), and (222), which appeared at the  $2\theta$  angles of  $51^\circ$ ,  $60^\circ$ ,  $91^\circ$ ,  $114^\circ$ , as well as  $122^\circ$ , respectively. The austenite diffraction showed distinctive pattern and had different scattering plane from ferrite diffraction pattern, which was body-centered cubic crystal system (Parikin et al., 2018). Moreover, the addition of  $Y_2O_3$  slightly changed diffraction patterns observable in peak shifting, intensity changing, and peak broadening. These effects were attributed to the variation of  $Y_2O_3$  dispersion in alloys. Figure 5 (b) showed that these effects also occurred in the primary peak (111), signifying changes in the lattice structure and strains of alloys.



**Figure 5** HRPD patterns of FeNiCr cast alloys with varying concentrations of  $Y_2O_3$ , showing peak intensities and positions. (a) HRPD patterns of FeNiCr cast alloys with  $Y_2O_3$  content ranging from 0 to 2.0 wt.% from  $2\theta$  at 40 to 130 degrees. Moreover, the peaks were sharp and distinct, with variations in intensity as well as position depending on the concentration of  $Y_2O_3$ . (b) A zoomed-in version of HRPD patterns focused on a specific range of  $2\theta$  angles from around 51.0 to 53.0 degrees.



These data showed the effect of  $Y_2O_3$  on changing lattice parameters of FeNiCr cast alloys, which were also strongly related to residual stress.

### 3.2.2. Refined parameters

Table 4 showed the refined parameters, namely, lattice, mass, and volume fractions of the phases. The mass fraction of cubic  $Y_2O_3$  in the specimens increased with its addition to the composition, while the volume fraction of  $Y_2O_3$  appeared to be randomly computed due to the separation of materials. All neutron diffraction profiles of the specimens were successfully refined with a goodness of fit (GOF) reliability ranging from 1.16 to 1.48.

**Table 4** Refined ambient-temperature lattice parameters and phase fractions of ODS FeNiCr $Y_2O_3$  specimens

Specimen Code	$Y_2O_3$ (wt.%)	GOF (%)	Lattice Parameters (Å)		Mass Fraction (w%)		Volume Fraction (f%)	
			FeNiCr (Austenite Matrice)	$Y_2O_3$ (Cubic-Yttria)	FeNiCr (Austenite Matrice)	$Y_2O_3$ (Cubic-Yttria)	FeNiCr (Austenite Matrice)	$Y_2O_3$ (Cubic-Yttria)
FeNiCr	-	1.20	3.59783	-	100.00	-	1.00000	-
FeNiCr-03 $Y_2O_3$	0.3	1.16	3.59069	10.61221	99.65	0.35	0.99865	0.00135
FeNiCr-05 $Y_2O_3$	0.5	1.19	3.59529	11.39172	99.41	0.59	0.99522	0.00478
FeNiCr-07 $Y_2O_3$	0.7	1.22	3.59929	10.55049	99.30	0.70	0.99367	0.00633
FeNiCr-10 $Y_2O_3$	1.0	1.41	3.59746	10.59284	98.82	1.18	0.99968	0.00324
FeNiCr-15 $Y_2O_3$	1.5	1.46	3.60936	10.65735	98.48	1.52	0.98937	0.01063
FeNiCr-20 $Y_2O_3$	2.0	1.48	3.67214	10.79821	97.93	2.06	0.96686	0.03314
$Y_2O_3$	100.0	1.40	-	11.48965	-	100.00	-	1.00000

### 3.2.3. Strain tensor properties

The refinements produced GOF with  $\sigma$  between 1.16 and 1.48 to diffraction data for each specimen. The experimental precision identified that the volume-averaged lattice strains along various [hkl] agreed with the crystal symmetry. This process was expected for the strains arising solely from the mismatch of thermal expansion in a mixture of randomly oriented grains. Since the strain tensor conformed to crystal symmetry, the number of independent components reduced to one. When analysis uniaxial crystal structures, such as in austenite matrices and  $Y_2O_3$  structures, an independent component sufficed to represent the strain tensor properties completely (Newnham, 2020). Along any other direction, the strains were computed using the tensor algebra (De Prunelé et al., 2007). The process was desirable to determine strain components along principal axes, which were the a-axis for austenite matrix phase and cubic  $Y_2O_3$  phase in conventional structure setting.

### 3.2.4. Thermal expansion mismatch

Table 5 showed that austenite matrix and FeNiCr $Y_2O_3$  ODS cast alloys were initially in a compression and then gradually in a tension after exceeding a composition of 1.0 wt.%  $Y_2O_3$ . Meanwhile,  $Y_2O_3$  phase signified compressive tendency for entire composition.

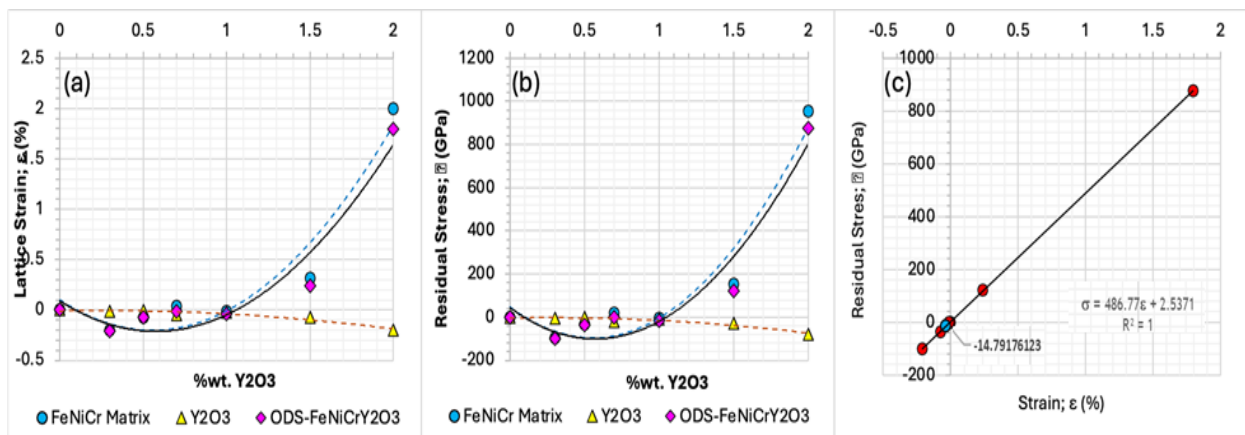
The strains measured in each phase were highly isotropic and stretching in materials was dominated by strain behavior of matrix (FeNiCr) phase. These features were qualitatively explained by considering thermal expansion mismatch between austenite matrix and  $Y_2O_3$  particles exchanging expansion when heating was applied during smelting (Kim et al., 2019; Koh et al., 2013).

**Table 5** Average lattice strains and hydrostatic stress along major crystallographic directions (*a*-axis) of the ODS FeNiCrY<sub>2</sub>O<sub>3</sub> specimens

Specimen Code	Average Lattice Strain, $\epsilon$ (%)			Hydrostatic Stress, $\sigma$ (GPa)
	<i>a</i> // FeNiCr (Austenite Matrice)	<i>a</i> // Y <sub>2</sub> O <sub>3</sub> (Cubic Yttria)	ODS FeNiCrY <sub>2</sub> O <sub>3</sub> ( <i>a</i> // resultant)	
FeNiCr	0	0	0	0
FeNiCr-03Y <sub>2</sub> O <sub>3</sub>	-0.198185045	-0.010309661	-0.208494706	-98.80982917
FeNiCr-05Y <sub>2</sub> O <sub>3</sub>	-0.070260652	-0.004074148	-0.0743348	-35.19377829
FeNiCr-07Y <sub>2</sub> O <sub>3</sub>	0.040323145	-0.0517412	-0.011418055	-0.918724427
FeNiCr-10Y <sub>2</sub> O <sub>3</sub>	-0.010280686	-0.025289407	-0.035570093	-14.79176123
FeNiCr-15Y <sub>2</sub> O <sub>3</sub>	0.317064344	-0.077002772	0.240061572	121.5717199
FeNiCr-20Y <sub>2</sub> O <sub>3</sub>	1.996963909	-0.199434461	1.797529447	877.1952868

### 3.2.5. Lattice strain and residual stress analysis

Figure 6 showed the effect of Y<sub>2</sub>O<sub>3</sub> content on lattice strain and residual stress of FeNiCrY<sub>2</sub>O<sub>3</sub> ODS cast alloys. As shown in Figure 6 (a), lattice strain of FeNiCrY<sub>2</sub>O<sub>3</sub> ODS phase increased with higher Y<sub>2</sub>O<sub>3</sub> content, while latter remained unchanged. The outcome signified that Y<sub>2</sub>O<sub>3</sub> dispersion induced significant distortion in the lattice of FeNiCrY<sub>2</sub>O<sub>3</sub> ODS phase. The result of the process improved strength and hardness of alloys. Similarly, Figure 6(b) showed that residual stress of FeNiCrY<sub>2</sub>O<sub>3</sub> ODS phase also increased with higher Y<sub>2</sub>O<sub>3</sub> content, having no significant variation in Y<sub>2</sub>O<sub>3</sub> phases. This outcome signified that Y<sub>2</sub>O<sub>3</sub> dispersion generated a compressive residual stress in FeNiCrY<sub>2</sub>O<sub>3</sub> ODS phase, improving the resistance to any crack propagation and some fracture in alloys. Figures 6(a) and (b) showed that lattice strains and residual stress of ODS specimens varied inconsistently when Y<sub>2</sub>O<sub>3</sub> contents were less than 1.0 wt.%.



**Figure 6** Lattice strain and residual stress analysis of ODS FeNiCrY<sub>2</sub>O<sub>3</sub> cast alloys as a function of Y<sub>2</sub>O<sub>3</sub> content. The lattice strain and residual stress were calculated from the peak broadening of the patterns using Williamson-Hall method. (a) Average lattice strains along the major crystallographic directions (*a*-axis). (b) Residual stress along the major crystallographic directions (*a*-axis). (c) Equilibrium residual stress-strain curve of alloys.

Strain and stress remained unchanged when Y<sub>2</sub>O<sub>3</sub> contents were 1.0 wt.%. However, both increased when the addition of Y<sub>2</sub>O<sub>3</sub> was more than 1.0 wt.%. The stretching of parallel crystal lattices *a*// and *b*// were distorted to eliminate each other in addition to the morphology and elemental mapping in alloys being better dispersed. Both lattices approached zero when Y<sub>2</sub>O<sub>3</sub> content was 1.0 wt.% with a compressive residual stress of about 14.79 GPa. The compressive stress under compositional equilibrium predicted that 1.0 wt.% Y<sub>2</sub>O<sub>3</sub> was considered stable in FeNiCrY<sub>2</sub>O<sub>3</sub> ODS cast alloy, and it showed excellence for further production.

Figure 6 (c) showed the relationship between residual stress and lattice strain for each phase. A linear relationship was observed, signifying that residual stress and lattice strain were proportional to each other. In addition, the slope of line represented elastic modulus of the phase, which reflected the stiffness of materials (Parikin and Allen, 2015; Yang et al., 2023). FeNiCr10Y<sub>2</sub>O<sub>3</sub> ODS phase had its strain and residual stress almost zero. This outcome showed that optimum Y<sub>2</sub>O<sub>3</sub> content in 58Fe25Ni17Cr alloys was about 1.0 wt.%. Among the four alloys containing Y<sub>2</sub>O<sub>3</sub> oxide signifying compressive residual stress behavior, the most moderate type recommended for further fabrication as FeNiCrY<sub>2</sub>O<sub>3</sub> ODS system was 57Fe25Ni17Cr1.0Y<sub>2</sub>O<sub>3</sub> cast alloy.

#### 4. Conclusions

In conclusion, this research investigated the effect of Y<sub>2</sub>O<sub>3</sub> dispersion on the micro-crystal structure and residual stress of FeNiCrY<sub>2</sub>O<sub>3</sub> ODS cast alloys. Alloys were synthesized by mechanical alloying and arc melt-casting methods as well as characterized by neutron diffraction and various microscopy methods. The results showed that Y<sub>2</sub>O<sub>3</sub> dispersion induced a lattice compression in austenitic FeNiCr matrix, which implied a tensile residual stress in the crystals. Moreover, the distribution of residual stress in alloys was related to individual mechanical properties and performance. Optimum Y<sub>2</sub>O<sub>3</sub> content in this type of FeNiCrY<sub>2</sub>O<sub>3</sub> ODS cast alloys was estimated to be about 1.0 wt.%, which showed better nano-dispersion and signified lowest strain and residual stress. The compressive stress of 14.79 GPa at strain equilibrium ( $\epsilon \sim 0$ ) was considered to be stable in this ODS cast alloys and excellent for further production in 57Fe25Ni17Cr1.0Y<sub>2</sub>O<sub>3</sub> system.

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#### Conflict of Interest

There are no conflicts of interest to disclose.

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