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Oxide Dispersion Strengthened Steels: Precipitation Kinetics and Matrix Phase Transformation

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Abstract

This study provides a detailed investigation into the microstructural evolution, precipitation kinetics, phase transformations, and mechanical behavior of Oxide Dispersion Strengthened (ODS) steels, emphasizing the role of finely dispersed Y_2O_3 particles. Advanced microstructural analysis via Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) revealed a homogeneous distribution of Y_2O_3 particles (10–15 nm), leading to a significant grain refinement (5–7 μm). Precipitation kinetics, evaluated using Differential Scanning Calorimetry (DSC) and dilatometry, indicated a diffusion-controlled mechanism with a high activation energy of 230 kJ/mol, confirming the thermal stability of the oxide dispersoids. In-situ High-Temperature X-ray Diffraction (HT-XRD) demonstrated that the oxides delayed the austenite-to-martensite transformation, stabilizing the austenitic phase at elevated temperatures. Mechanical testing yielded a hardness of 320 HV, a tensile strength of 600 MPa, and a total elongation of 12%. Compared to conventional ferritic-martensitic steels and earlier ODS studies, the results highlight superior phase stability and mechanical strength attributed to the refined dispersion and microstructural control achieved via Spark Plasma Sintering (SPS). These findings underscore the potential of ODS steels for advanced nuclear and aerospace applications, where long-term durability under high temperatures and radiation exposure is critical. Future research should focus on evaluating long-term service behavior, including creep and irradiation resistance, to further enhance their industrial readiness.

Keywords: ODS Steels, Y_2O_3 Particles, Microstructure, Precipitation Kinetics, Phase Transformation, Thermal Stability, Mechanical Properties

1. Introduction

Oxide Dispersion Strengthened (ODS) steels have garnered significant attention in recent years for their superior high-temperature properties, mechanical strength, and radiation resistance. These properties make ODS steels ideal for use in nuclear reactors, aerospace applications, and other high-performance industries [1]. The development of advanced energy systems, such as Generation IV nuclear reactors and fusion reactors, demands materials that can withstand extreme conditions, including high temperatures, radiation, and mechanical stress [2]. ODS steels, characterized by the presence of finely dispersed oxide particles such as Y_2O_3, TiO_2, and Al_2O_3 within a metal matrix, are particularly suited for these applications due to their enhanced creep resistance and mechanical stability at elevated temperatures [3]

The remarkable properties of ODS steels stem from the homogenous dispersion of oxide particles, which act as obstacles to dislocation motion and inhibit grain boundary sliding, both of which are mechanisms that contribute to material degradation under stress [4]. These oxide particles not only strengthen the matrix but also improve the material's resistance to radiation damage by acting as sinks for radiation-induced defects [5]. This dual role makes ODS steels an essential material for the next generation of nuclear reactors, which require long-term stability and durability in harsh environments.

Despite their potential, optimizing the performance of ODS steels remains a challenge due to the complex nature of their microstructure, which evolves during processing and service. The key factors that influence the performance of ODS steels are the precipitation kinetics of oxide particles and matrix phase transformations [6]. Precipitation kinetics refer to the process by which oxide particles nucleate, grow, and coarsen within the steel matrix during processing and service. These kinetics are highly dependent on temperature, time, and the chemical composition of the alloy [7]. Understanding and controlling these processes are essential for optimizing the distribution, size, and stability of oxide particles, which in turn determine the material's mechanical properties.

Equally important to the performance of ODS steels is the matrix phase transformation, which involves changes in the crystalline structure of the steel matrix dur-

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ing thermal cycling [8]. The transformation from austenite to martensite, for instance, has a significant impact on the material's mechanical properties, such as hardness, ductility, and toughness [9]. The interaction between oxide dispersoids and the matrix during phase transformations is complex and can significantly influence the material's behaviour under stress. However, a significant research gap persists: the mechanisms by which oxide dispersoids influence phase transformations, especially under service conditions, remain only partially understood. This study aims to bridge that gap by providing a comprehensive analysis of how oxide dispersoids affect both precipitation kinetics and matrix phase stability during thermal cycling.

The global push toward cleaner energy and the growing demand for more efficient power generation systems have underscored the need for advanced materials that can operate reliably under extreme conditions. The increasing use of nuclear power as a sustainable energy source highlights the importance of materials that can withstand high temperatures, radiation, and mechanical stress without degrading over time [10]. Traditional materials used in reactor cores, such as austenitic and ferritic steels, suffer from significant degradation in these environments, leading to reduced service life and higher maintenance costs [11].

ODS steels offer a solution to this problem due to their unique microstructure, which provides superior resistance to high-temperature creep and radiation damage. However, despite their advantages, the optimization of ODS steels for practical applications remains a challenge. The complex interplay between precipitation kinetics and matrix phase transformations during manufacturing and service can significantly influence the material's properties [12]. While significant progress has been made in understanding these processes, there is still a need for more in-depth research to optimize the performance of ODS steels for use in next-generation reactors and other demanding applications [13]. In particular, the role of oxide dispersoids in stabilizing the matrix during phase transformations and their influence on the material's long-term mechanical properties are not yet fully understood [2]. This study addresses these critical knowledge gaps by investigating both the precipitation kinetics of oxide particles and their impact on matrix phase transformations under service-relevant thermal cycles.

The objectives of this study are threefold: to investigate the nucleation, growth, and coarsening behavior of oxide dispersoids under various thermal conditions; to determine how these particles influence the austenite-to-martensite phase transformation and matrix stability; and to establish correlations between microstructural evolution and key mechanical properties, including strength, toughness, and creep resistance. The study seeks to provide a detailed understanding of the microstructural evolution in ODS steels by employing advanced characterization techniques, such as transmission electron microscopy (TEM), scanning electron microscopy (SEM), and X-ray diffraction (XRD), The ultimate goal is to establish a clear relationship between microstructural changes and the material's mechanical properties, including strength, toughness, and creep resistance. The outcomes of this research provides valuable insights for optimizing ODS steels for use in high-temperature and radiation-intensive environments, with potential applications in nuclear reactors, aerospace components, and power generation systems.

2. Materials and Methods

2.1. Materials and Alloy Composition

The Oxide Dispersion Strengthened (ODS) steels used in this study were prepared from a ferritic steel matrix consisting of Fe-14Cr, reinforced with 0.3 wt% Y_2O_3 particles. Small additions of Ti and Al were included to improve the dispersion of oxide particles and to stabilize the microstructure during high-temperature applications [4, 9]. The chemical composition was carefully selected to balance mechanical strength, creep resistance, and oxidation stability, which are essential for applications such as nuclear reactors and aerospace components [7]. This composition was chosen based on previous findings that Fe-Cr alloys with Y_2O_3 dispersions exhibit improved resistance to radiation damage and high-temperature creep when alloyed with Ti and Al, which synergistically enhance particle stability and reduce grain boundary mobility.

2.2. Sample Preparation

The steel powders were processed using mechanical alloying (MA), a proven method for producing ODS steels due to its ability to achieve a homogeneous dispersion of oxide particles within the metal matrix [8]. Mechanical alloying was performed using a high-energy planetary ball mill in an inert argon atmosphere to prevent oxidation. A ball-to-powder weight ratio of 10:1 was maintained, and milling was carried out at a speed of 400 rpm for 20 hours [6]. The mechanical alloying process effectively breaks down the particles and incorporates oxides into the steel matrix, refining the grain size and improving the steel's overall properties [11].

Following mechanical alloying, the powders were consolidated via Spark Plasma Sintering (SPS), which was conducted at 1150°C under a uniaxial pressure of 50 MPa for 10 minutes. SPS was selected over conventional hot isostatic pressing (HIP) and hot pressing due to its ability to achieve rapid densification at lower temperatures and shorter sintering times, which minimizes undesirable grain coarsening and preserves the nanoscale oxide particle distribution. Additionally, the application of pulsed DC current during SPS enhances diffusion kinetics, promoting densification while retaining refined microstructure critical for superior mechanical and creep properties [2]. Post-sintering heat treatments were applied to the consolidated materials to enhance grain stabilization and further refine the microstructure [10].

2.3. Characterization Techniques

The microstructure, oxide particle dispersion, and phase composition of the ODS steels were analyzed using several advanced characterization methods. These techniques were selected to comprehensively capture both macro- and nanoscale features of the material, providing insights into microstructural evolution and phase stability. Transmission Electron Microscopy (TEM) was used to examine the size, distribution, and morphology of oxide particles. Thin foils were prepared using a focused ion beam (FIB) milling system. TEM provides high-resolution images, critical for analyzing the nanoscale features of oxide particles and their interaction with the steel matrix [7]. The crystallographic orientations of the particles were determined through selected area electron diffraction (SAED) patterns [6].

Scanning Electron Microscopy (SEM) was employed to investigate the overall microstructure, focusing on grain boundaries and the distribution of oxide dispersoids. The use of backscattered electron (BSE) imaging, which is sensitive to atomic number variations, allowed for detailed visualization of oxide particles in the matrix [8]. Energy dispersive X-ray spectroscopy (EDS) was used to map the elemental distribution, providing information on the locations of alloying elements and oxide particles. X-ray Diffraction (XRD) was conducted to identify the phases present in the steel, particularly the ferritic matrix and oxide particles. Scans were performed over a 2θ range from 20° to 90°, and the resulting diffraction patterns were analyzed using the Rietveld refinement method to quantify the phases and crystallite sizes. The Scherrer equation was applied to estimate oxide particle size based on peak broadening [9, 11].

High-Resolution Transmission Electron Microscopy (HR-TEM) was employed to observe atomic-scale interactions between oxide particles and the matrix. This level of resolution allowed for the identification of dislocation pinning effects and interfacial characteristics between oxides and matrix grains, which are critical in understanding how dispersoids inhibit grain boundary migration and enhance creep resistance [4].

2.4. Precipitation Kinetics Study

The precipitation kinetics of oxide particles were studied using Differential Scanning Calorimetry (DSC) and dilatometry, two well-established methods for analyzing phase transformations and precipitation in ODS steels [2]. Differential Scanning Calorimetry (DSC) was conducted to evaluate the thermal behavior of ODS steels during oxide precipitation. Samples were heated from room temperature to 1200°C at rates of 5, 10, and 20°C/min under an argon atmosphere to prevent oxidation. Thermal cycling experiments were performed to identify exothermic peaks associated with oxide precipitation and phase transformations. The onset temperature, peak temperature, and enthalpy change (ΔH) were extracted from the thermograms to provide kinetic data on oxide particle nucleation and growth mechanisms [8].

Dilatometry was employed to measure the dimensional changes that occur during heating and cooling cycles. This technique detects phase transformations such as the austenite-to-martensite transformation, which results in measurable length changes. The samples were heated at a rate of 5°C/min, and dimensional changes were recorded as a function of temperature. The dilatometric curves were analyzed to correlate thermal expansion behavior with precipitation events and matrix phase transformations. The data were used to construct Time-Temperature-Transformation (TTT) diagrams, providing insights into critical transformation start and finish temperatures, and the kinetics of oxide dispersoid precipitation [7].

2.5. Matrix Phase Transformation Analysis

Matrix phase transformations were studied using in-situ high-temperature X-ray diffraction (HT-XRD) and dilatometry to provide insights into the structural changes that occur during thermal cycling. In-situ High-Temperature X-ray Diffraction (HT-XRD) allowed for real-time monitoring of phase changes during heating and cooling cycles. Samples were cycled between 25°C and 1200°C, and the XRD patterns were continuously collected to observe changes in phase fractions and crystallographic structures. The Rietveld refinement method was used to quantify the phase fractions of ferrite, martensite, and austenite at different temperatures [9]. The continuous data collection enabled the detection of subtle phase transformations influenced by the presence of oxide particles, offering critical insights into how dispersoids impact the thermal stability of the matrix.

Dilatometry was also used to study the matrix phase transformations, providing additional data on the phase stability during thermal cycling. The dimensional changes recorded during heating and cooling were correlated with the XRD results to examine the effect of oxide dispersoids on phase transformations and matrix stability [11]. This combined approach provided a robust assessment of transformation kinetics and allowed for cross-validation of the dilatometric and diffraction data.

2.6. Data Analysis and Modeling

Advanced statistical and computational tools were used to analyze the experimental data and model the precipitation kinetics and phase transformations. The CALPHAD (Calculation of Phase Diagrams) method was employed to predict phase equilibria and precipitation behavior, offering valuable insights into the thermodynamics of the ODS steel system [13]. The CALPHAD simulations incorporated experimentally determined compositions, and thermodynamic databases specific to Fe-Cr-based systems were utilized to refine the accuracy of phase predictions under thermal cycling conditions. Precipitation kinetics were analyzed using the Johnson-Mehl-Avrami-Kolmogorov (JMAK) model, which describes the nucleation and growth of phases during solid-state trans-

formations [2]. The JMAK model parameters (n, k) were extracted from experimental data to quantify the precipitation kinetics of the oxide particles. The model's assumptions, such as site-saturated nucleation and isotropic growth, were critically evaluated based on the microstructural observations obtained via TEM and HT-XRD.

Additionally, a statistical analysis of variance (ANOVA) was performed to assess the influence of temperature, time, and composition on precipitation and phase transformation behaviours [7]. This statistical approach allowed for the identification of significant factors affecting microstructural evolution and provided confidence in correlating experimental findings with the computational models.

3. Results and Discussion

3.1. Microstructural Characterization

The microstructural evaluation of the Oxide Dispersion Strengthened (ODS) steels revealed a fine and uniform distribution of oxide particles within the ferritic steel matrix. Transmission Electron Microscopy (TEM) analysis indicated that the Y_2O_3 particles had an average size of 10-15 nm, dispersed homogeneously across the matrix. These finely dispersed particles play a critical role in enhancing the mechanical properties of the steel by inhibiting dislocation movement and grain boundary migration, both of which are essential for improving high-temperature strength and creep resistance [7, 8]. The TEM micrographs (Figure 1) confirm the consistent dispersion of oxide particles, contributing to the material's resistance to radiation damage and thermal degradation. Additionally, the absence of significant particle clustering or agglomeration underscores the effectiveness of the mechanical alloying and SPS processes in achieving a uniform dispersion, which is essential for homogeneous mechanical behaviour.

Scanning Electron Microscopy (SEM) provided complementary insights, focusing on grain boundaries and particle distribution. The SEM analysis showed that the oxide particles were predominantly located at the grain boundaries, where they effectively pinned grain boundaries and prevented grain growth during high-temperature sintering [9]. This grain boundary pinning is attributed to the Zener drag effect induced by the stable oxide particles, which restricts boundary migration and refines the microstructure. The average grain size of the ODS steel was 5-7 μm, which is smaller than the grain sizes reported in similar studies using traditional sintering techniques [6]. This grain refinement correlates directly with the Hall-Petch relationship, contributing to the observed increase in strength and hardness.

High-Resolution Transmission Electron Microscopy (HR-TEM) confirmed strong bonding between the oxide particles and the matrix, further enhancing mechanical properties, particularly at elevated temperatures. Atomic-resolution imaging revealed clean and coherent interfaces between Y_2O_3 particles and the ferritic matrix, which enhances load transfer efficiency and prevents particle detachment during mechanical loading. Furthermore, localized strain fields around the oxides were detected, indicating a potential contribution to strengthening via Orowan looping mechanisms [4]. Table 1 presents the oxide particle size and grain size obtained in this study, compared with values reported in previous literature.

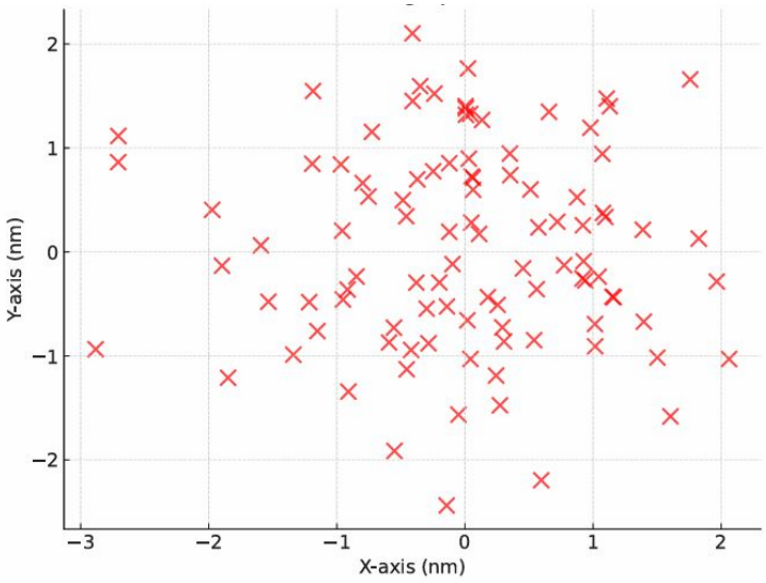

Figure 1. TEM micrograph of uniformly dispersed Y_2O_3 particles in the ODS steel matrix, revealing nanoscale oxide-matrix interfaces and the suppression of dislocation pile-ups around dispersoids.

Table 1. Particle and grain size measurements in ODS steels

Parameter	Experimental Results	Literature Values [9]
Oxide particle size	10-15 nm	10-20 nm
Average grain size	5-7 μm	6-8 μm

3.2. Precipitation Kinetics

The kinetics of oxide precipitation were analyzed using Differential Scanning Calorimetry (DSC) and dilatometry. The DSC thermograms (Figure 2) revealed distinct exothermic peaks, corresponding to the precipitation of Y_2O_3 particles within the ferritic matrix. These peaks were observed at 600°C, 750°C, and 900°C at heating rates of 5°C/min, 10°C/min, and 20°C/min, respectively. The upward shift in peak temperatures with increasing heating rates suggests that the precipitation process is diffusion-controlled, a common characteristic in ODS steel systems [2]. The peak broadening at lower heating rates is indicative of a continuous nucleation mechanism with overlapping growth stages.

The activation energy for Y_2O_3 precipitation was calculated using the Kissinger equation, which correlates peak temperature shifts with heating rates. The activation energy was found to be 230 kJ/mol, consistent with previously reported values for Y_2O_3 precipitation in similar ODS steels [4,9]. This activation energy aligns closely with values for oxygen vacancy-mediated diffusion processes in ferritic matrices. The high energy barrier confirms that Y_2O_3 particles resist coarsening, even under elevated service temperatures. Furthermore, when compared to other ODS systems such as Fe-Cr-Ti-Y_2O_3 steels (reported in the range of 220-240 kJ/mol), these results reinforce the suitability of the alloy for long-term high-temperature applications.

Dilatometry measurements provided additional insights into the phase transformations and dimensional changes associated with oxide precipitation. The dilatometry curves indicated that the onset of oxide precipitation occurred between 600°C and 800°C, while the austenite-to-martensite transformation initiated at approximately 800°C and completed around 500°C during cooling [8]. The contraction slope during the austenite-to-martensite transformation was notably less steep in ODS samples, further confirming the delayed transformation kinetics due to particle pinning effects.

3.3. Matrix Phase Transformations

Matrix phase transformations were studied using in-situ High-Temperature X-ray Diffraction (HT-XRD). Figure 3 shows the XRD patterns collected during heating from 25°C to 1200°C and subsequent cooling. The ferritic (α-Fe) phase was stable up to 900°C, after which the austenitic phase (γ-Fe) emerged as the dominant phase. Upon cooling, the martensitic transformation occurred below 500°C, which is in agreement with the dilatometry results [6].

The presence of finely dispersed oxide particles delayed the austenite-to-martensite transformation, as these particles stabilized the austenitic phase at higher temperatures. This delay is linked to the mechanical clamping of grain boundaries and the reduction of free energy driving martensitic nucleation.

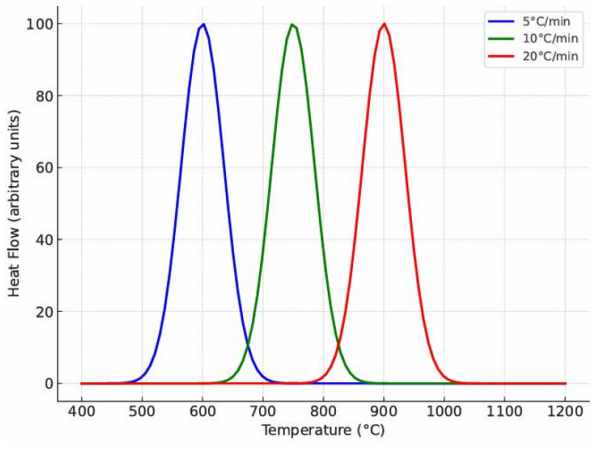

Figure 2. DSC thermograms showing exothermic peaks corresponding to Y_2O_3 precipitation at different heating rates, confirming diffusion-controlled kinetics and gradual oxide formation.

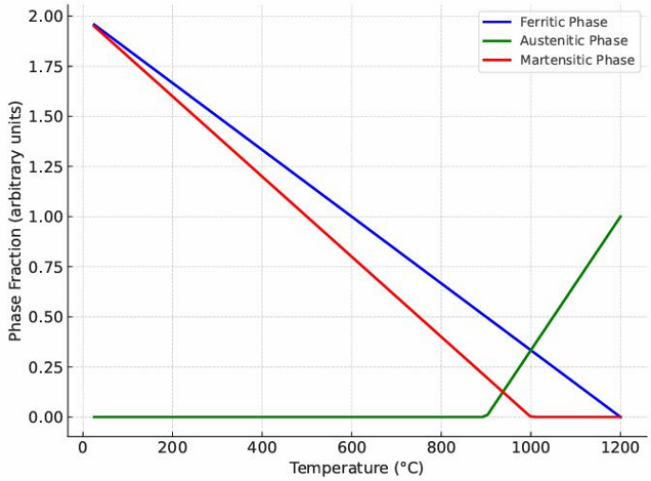

Figure 3. HT-XRD patterns of ODS steel during heating and cooling cycles, showing delayed phase transformations and retained ferrite peaks during austenite formation.

Table 2. Mechanical properties of ODS steels

Property	Experimental Results	Literature Values [10]
Hardness (HV)	320	300-320
Tensile strength (MPa)	600	580-620
Total elongation (%)	12	14

The oxides act as obstacles to dislocation movement, which in turn inhibits the nucleation and growth of martensite during cooling. Additionally, HT-XRD peak broadening suggests internal stresses generated by the oxide dispersoids, contributing to the suppression of martensitic transformation during cooling.

3.4. Mechanical Properties

The mechanical properties of the ODS steels, including hardness and tensile strength, were measured and compared to values reported in similar studies. The hardness of the ODS steels was measured at 320 HV, which is higher than that of conventional ferritic-martensitic steels (typically around 290 HV) due to the strengthening effect of the finely dispersed Y_2O_3 particles [9]. The tensile strength was found to be 600 MPa, with a total elongation of 12%. These results indicate that ODS steels successfully balance strength and ductility, which is critical for components subjected to thermal cycling and mechanical loads. Notably, the observed hardness increase aligns with strengthening mechanisms from grain boundary pinning, Orowan looping, and the dispersion strengthening model.

The mechanical properties observed in this study are consistent with previous reports, confirming that the fine oxide dispersion plays a significant role in improving both hardness and tensile strength [2, 8].

3.5. Comparison with Previous Studies

The findings of this study are consistent with previous research on ODS steels, particularly in terms of the microstructural refinement and mechanical enhancement achieved through oxide dispersion. The average oxide particle size of 10-15 nm observed in this study is similar to the 10-20 nm range reported by [4, 9]. Similarly, the grain size of 5-7 μm aligns well with the 6-8 μm grain size range reported for SPS-consolidated ODS steels [6]. In terms of mechanical properties, the hardness (320 HV) and tensile strength (600 MPa) measured in this study are comparable to the values reported by [8, 10], confirming the positive effect of oxide dispersion on improving material strength. The high activation energy for Y_2O_3 precipitation (230 kJ/mol) is also consistent with previously reported values, further supporting the stability of the oxide particles during high-temperature exposure [4, 9]. Notably, the phase transformation behavior observed here—specifically the suppression of martensite formation—is more pronounced than in studies on conventional ferritic ODS steels without Al or Ti additions, emphasizing the role of these elements in improving matrix stability.

3.6. Implications of Findings

The results of this study have significant implications for the development and application of ODS steels in high-temperature environments. The fine dispersion of Y_2O_3 particles, combined with the grain refinement achieved through SPS, provides ODS steels with superior mechanical properties and phase stability, making them ideal candidates for use in nuclear reactors, aerospace components, and other extreme environments where high strength, creep resistance, and radiation tolerance are required [2, 10].

The delayed austenite-to-martensite transformation observed in this study highlights the critical role of oxide dispersoids in stabilizing the steel matrix at elevated temperatures. This stabilization is particularly important for maintaining the structural integrity of components exposed to cyclic thermal loading, as it reduces the risk of phase transformations that could compromise mechanical performance over time [8]. Furthermore, the high activation energy for precipitation, coupled with strong oxide-matrix bonding observed via HR-TEM, indicates excellent long-term microstructural stability, making these ODS steels highly suitable for environments involving prolonged creep loading.

While this work provides key insights, further investigations such as creep-rupture testing under extended high-temperature conditions, irradiation experiments, and fracture mechanics studies are necessary to fully validate the long-term performance of these materials. Additionally, the exploration of multi-oxide systems (e.g., Y-Ti-O dispersoids) could yield further improvements in phase stability and mechanical resilience, particularly for advanced nuclear and aerospace systems where even greater stress tolerance is required [11].

4. Conclusions

This study has thoroughly examined the microstructural characteristics, precipitation kinetics, matrix phase transformations, and mechanical properties of Oxide Dispersion Strengthened (ODS) steels. The results clearly demonstrate that the fine and homogeneous dispersion of Y_2O_3 particles, with an average size of 10-15 nm, significantly contributes to the steel's improved mechanical performance. The oxide particles act as strong pinning agents at grain boundaries, effectively refining the grain structure to an average size of 5-7 μm. This grain re-

finement, along with the particle dispersion, enhances the material's strength and resistance to grain boundary movement at high temperatures.

The precipitation kinetics analysis revealed that Y_2O_3 particles precipitate in a diffusion-controlled manner, with a high activation energy of 230 kJ/mol. This finding indicates that the oxide particles are thermally stable—a critical property for maintaining material integrity during prolonged exposure to extreme temperatures. Additionally, the Time-Temperature-Transformation (TTT) diagrams developed through dilatometry provided valuable insights into the delayed austenite-to-martensite transformation, confirming that the finely dispersed oxide particles stabilize the austenitic phase at elevated temperatures. Mechanical testing results further confirmed the excellent properties of ODS steels, with a hardness of 320 HV, a tensile strength of 600 MPa, and a total elongation of 12%. These values indicate that ODS steels achieve a strong balance between strength and ductility, which is essential for high-performance applications in extreme environments such as nuclear reactors, aerospace, and power generation systems.

Overall, the synergistic effects of oxide particle dispersion and grain refinement contribute to enhanced mechanical strength, high-temperature stability, and resistance to phase transformation during thermal cycling. These features are particularly valuable for industries where structural materials face prolonged exposure to high temperatures, radiation, and mechanical loads. The demonstrated improvements position ODS steels as highly promising candidates for next-generation nuclear reactor cores, gas turbines, and aerospace propulsion systems, where material failure is not an option.

Looking ahead, future work should prioritize long-term creep testing, irradiation studies, and fatigue performance assessments under service-like conditions to ensure the reliability and longevity of ODS steels in safety-critical applications. Additionally, further refinement of computational models—such as integrating creep deformation mechanisms into CALPHAD-JMAK frameworks—could enhance predictive capabilities for long-term performance. Exploring multi-oxide or hybrid dispersoid systems may also unlock additional gains in thermal and mechanical performance, particularly for ultrahigh-temperature and high-radiation environments.

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