Microstructural Stability of Y2O3 Dispersion-Strengthened Low-Activation Steel (Postprint)

Authors: Hu Xue, Huang Lixin, Yan Wei, Wang Wei, Shan Yiyin, Yang Ke

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Abstract

CLAM steel mixed powder doped with 0.3% Y2O3 was prepared by mechanical alloying and consolidated by hot isostatic pressing (HIP) to fabricate ODS-CLAM steel with a density as high as 98.7%. Investigation of the morphology, distribution of alloying elements, and lattice distortion of the ball-milled CLAM steel alloy powder using secondary electron imaging, electron probe microanalysis, and X-ray diffraction (XRD) techniques determined the optimal ball milling process for the ODS-CLAM steel mixed powder to be: ball milling time of 50 h, ball-to-powder ratio of 10:1, hard steel balls with a diameter of 6 mm as milling media, and Ar atmosphere. The ODS-CLAM steel prepared using this process exhibited good high-temperature microstructural stability; after solution treatment at 1200 ℃ for 60 min, its grain size did not coarsen significantly, the laths remained fine, and the Y2O3 particles were still stably present in the matrix.

Full Text

Study on Microstructure Stability of a Y_2O_3 Dispersion Strengthened Low-Activation Steel

HU Xue[1],[2]), HUANG Lixin[3]), YAN Wei[1]), WANG Wei[1]), SHAN Yiyin[1]), YANG Ke[1])
[1]) Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016
[2]) University of Chinese Academy of Sciences, Beijing 100049
[3]) State Key Laboratory of Metastable Materials Science and Technology, Yanshan University, Qinhuangdao 066004
Correspondent: SHAN Yiyin, professor, Tel: (024)23971517, E-mail: yyshan@imr.ac.cn
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Abstract

Oxide dispersion strengthened (ODS) steels are being developed as promising structural materials for next-generation nuclear energy systems due to their excellent resistance to both irradiation damage and high-temperature creep. In this work, mechanical alloying (MA) and hot isostatic pressing (HIP) technologies were used to prepare an ODS low-activation steel based on China low activation martensitic (CLAM) steel. SEM, XRD analysis, and EPMA were used to examine the particle size, alloying element distribution, and lattice distortion of the ball-milled powders. To obtain uniform powders, CLAM powders with 0.3% Y_2O_3 particles should be milled with hard steel balls of 6 mm in diameter for 50 h in an Ar protective atmosphere, with a ball-to-powder weight ratio of 10:1. The microstructure of the well-prepared ODS-CLAM steel remained stable up to 1200 °C for 1 h, with a grain size of 50–60 μm and martensitic lath width of 200 nm; meanwhile, the Y_2O_3 particles could still be found in the steel matrix.

KEY WORDS oxide dispersion strengthened steel; microstructure; microstructure stability; Y_2O_3

Introduction

As the global energy crisis intensifies, developing nuclear fusion energy has become one of the important solutions. However, the service environment in fusion reactors is extremely harsh, requiring structural materials with superior performance. Low-activation ferritic/martensitic steels have become important candidate structural materials for future fusion reactors due to their low activation characteristics, high strength, good weldability, and microstructural stability. Reduced activation ferritic/martensitic (RAFM) steels replace conventional activation-prone alloying elements such as Mo, Nb, and Ni with low-activation elements like 1–2% W, 0.02–0.18% Ta, and approximately 0.2% V (mass fraction). This reduces the half-life of induced radioactive nuclides produced by (n,α) transmutation during irradiation to several hundred days, significantly shortening the storage period of decommissioned fusion reactor structural materials.

However, the applicable temperature range for RAFM steels is only 250–550 °C. When RAFM steels are exposed to temperatures exceeding 550 °C for extended periods, the martensitic laths in their microstructure fragment into substructures, and $M_{23}C_6$-type carbides coarsen significantly. Additionally, despite their low-activation properties, RAFM steels still undergo (n,α) reactions under high-dose neutron irradiation, generating He atoms that gradually accumulate and form He bubbles, leading to a significant increase in the ductile-to-brittle transition temperature. These weaknesses limit the application of RAFM steels in future fusion reactors, making it crucial to improve their microstructural stability and irradiation resistance.

To further enhance the microstructural stability and irradiation resistance of RAFM steels, preparing oxide dispersion strengthened (ODS) low-activation steel via powder metallurgy is a viable approach, extending their applicable temperature range to 250–650 °C. Currently, internationally studied ODS low-activation steels include MA957, 14YWT, and ODS-Eurofer. MA957 and 14YWT incorporate Ti and Y_2O_3 particles into Fe-Cr-Mo/W matrices. Sakasegawa et al. demonstrated that the addition of 1.0% Ti and 0.25% Y_2O_3 in MA957 steel leads to chemical reactions, generating numerous non-stoichiometric Y-Ti-O nanoclusters ($Y/Ti < 1$) of 2–15 nm, $Y_2Ti_2O_7$ particles of 15–35 nm, and a small number of TiO_2 particles larger than 35 nm. ODS-Eurofer steel, on the other hand, is developed from Eurofer steel with added Y_2O_3 particles. Cayron et al. proposed that Y_2O_3 particles decompose during ball milling and dissolve diffusively into Eurofer steel powder. As Y_2O_3 content increases, the alloy powder strength rises, and powder fragmentation becomes more severe, resulting in ODS-Eurofer steel powder with 1.0% Y_2O_3 having a much smaller particle size (~20 μm) than that with 0.2% Y_2O_3 (~50 μm). Lucon investigated the effect of Y_2O_3 content on the strength and toughness of ODS-Eurofer steel, finding that the tensile strength of ODS-Eurofer steel with 0.5% Y_2O_3 was about 100 MPa higher than that with 0.3% Y_2O_3, while the impact toughness of the two was similar. Klimenkov et al. observed that 5–10 nm particles in ODS-Eurofer steel exhibit a core-shell structure, with the shell primarily composed of V, Cr, and O (thickness 0.5–1.5 nm) and the core being $(Y_{1\cdot 8}Mn_{0\cdot 2})O_3$ phase with the same lattice parameters as bcc-Y_2O_3. Williams et al. suggested two theories to explain the formation of these nanoscale core-shell structured particles with V- and Cr-rich shells and Y-rich cores: first, V, Cr, and O elements segregate at the interface between nanoparticles and matrix during particle formation, creating V-, Cr-, and O-rich shells; second, the shell structure is a necessary condition for nanoparticle formation, acting as an interfacial phase that reduces surface energy and enables nanoparticle formation at the shell. Additionally, Williams et al. used 3D-AP to discover that the core of 5–10 nm shell-structured nanoparticles mainly contains 5–10% Mn, 5–10% Si, Y, and O, with the stoichiometric ratio of alloying elements to O in the core phase changing from approximately 1:2 to >1:1 as particle size increases. These dispersedly distributed nanoparticles ensure excellent properties in ODS steels. During high-dose neutron irradiation, the interfaces between nanoscale Y_2O_3 particles and matrix serve as He traps, preventing He atoms from aggregating into He bubbles. Under long-term service in high-temperature complex stress environments, inert nanoparticles can still pin dislocations and hinder dislocation glide, strengthening the alloy and giving ODS steel significantly higher high-temperature strength and creep performance than RAFM steels such as Eurofer97. Therefore, developing new ODS steel based on China’ s independently developed RAFM steel candidate for fusion reactor first walls is of great strategic significance.

Currently, China has independently developed two RAFM steels: CLAM (China Low Activation Martensitic steel), jointly developed by the Institute of Plasma Physics and Institute of Metal Research, Chinese Academy of Sciences; and CLF-1 (China Low-activation Ferritic steel), jointly developed by the Southwestern Institute of Physics and Institute of Metal Research, Chinese Academy of Sciences. This work prepares ODS-CLAM oxide dispersion strengthened low-activation steel using CLAM steel as the matrix and investigates the effect of Y_2O_3 particles on the high-temperature microstructural stability of ODS-CLAM steel, providing a research basis for subsequent development and practical application of ODS-CLAM steel.

1. Experimental Methods

The chemical compositions of CLAM steel and ODS-CLAM steel used in this work are shown in Table 1. The raw CLAM steel powder was prepared by: first, smelting CLAM steel ingots; then removing risers, surface oxide scale, and other impurities; and finally preparing powder by gas atomization. Specific atomization requirements: particle size < 50 μm, protective atmosphere of high-purity Ar gas, gas pressure > 3.5 MPa. The O and N contents in CLAM steel powder prepared by this atomization process were 0.023% and 0.12%, respectively. Under Ar inert atmosphere protection, raw CLAM steel powder (particle size 14.6 μm) and 0.3% Y_2O_3 powder (particle size 50 nm) were mixed and ball-milled for 50 h in a vertical ball mill. The milled powder was then loaded into a low-carbon steel capsule, which was sealed by welding. A small hole was reserved in the capsule lid, and a thin-walled steel tube with 3 mm inner diameter was welded on. The capsule was degassed at 400 °C under vacuum (10^{-1} Pa) for 4 h to remove adsorbed air from powder surfaces and improve material density. The mixed powder capsule was then subjected to hot isostatic pressing at 1200 °C and 150 MPa for 4 h, achieving a density of 98.7% in the consolidated bulk material. The HIP-consolidated ODS-CLAM steel was hot-rolled into 10 mm thick plate at 1200 °C (total deformation 73.7%). The final heat treatment for both CLAM and ODS-CLAM steels was: normalizing at 980 °C for 30 min, air cooling + high-temperature tempering at 760 °C for 90 min, air cooling. The room- and high-temperature tensile properties of CLAM and ODS-CLAM steels are shown in Table 2. Two tensile tests were conducted at room temperature and 600 °C for each material. CLAM steel tensile specimens were F5M10 rod samples with gauge dimensions of Ø5 mm × 25 mm; ODS-CLAM steel tensile specimens were F3M6 rod samples with gauge dimensions of Ø3 mm × 15 mm. The tensile properties of ODS-CLAM steel are comparable to similar ODS steels such as ODS-Eurofer and PM2000.

To investigate the high-temperature microstructural stability of ODS-CLAM steel, both CLAM and ODS-CLAM steels were solution-treated at 950–1200 °C for 30–60 min. Metallographic samples before and after solution treatment were etched with a solution of 1 g picric acid + 5 mL hydrochloric acid + 100 mL alcohol for 10–45 s.

An S-3400N scanning electron microscope (SEM) was used to observe alloy powder morphology; an EPMA-1610 electron probe microanalyzer was used to analyze the distribution of alloying elements in ball-milled powders; a D/max 2500PC X-ray diffractometer (Cu Kα radiation, tube voltage 50 kV, tube current 300 mA, graphite monochromator for diffracted beam monochromatization) was used to analyze lattice distortion in alloy powders; and an MEF-4A optical microscope (OM) and Tecnai20 transmission electron microscope (TEM) were used to observe grain size, martensitic microstructure morphology, and precipitate distribution in CLAM and ODS-CLAM steels before and after solution treatment.

2.1 Ball Milling Preparation of ODS-CLAM Steel

[Figure 1: see original paper] shows SEM secondary electron images of atomized CLAM steel powder and Y_2O_3 particles. The un-milled CLAM steel powder particles appear as regular spheres with an average size of approximately 14.6 µm (Fig. 1a). Y_2O_3 powder particles have an average size of about 50 nm and tend to agglomerate into clusters (Fig. 1b).

[Figure 2: see original paper] shows SEM images of CLAM steel powder with 0.3% Y_2O_3 addition after ball milling for different times. The powder particle size (D) after various milling times (t) was measured using the intercept method, as shown in [Figure 3: see original paper]. Fitting revealed the following relationship: $\lg D = 1.648 - 0.012t$. As seen in Figs. 2 and 3, powder refinement is significant in the initial milling stage. After 10–50 h of milling, CLAM steel powder particle size decreases from 32.0 µm to 9.6 µm; however, after 60 h of milling, the particle size shows no significant further reduction, remaining at approximately 8.7 µm. When 6 mm diameter hard steel balls collide violently with CLAM steel powder mixtures, the rapidly rotating balls force the powder to undergo plastic deformation, producing cold welding and fragmentation due to severe work hardening. The fresh fracture surfaces enable new cold welding and atomic diffusion. This repeated cycle of cold welding-fragmentation-re-cold welding-re-fragmentation gradually reduces CLAM steel powder particle size while increasing defect density, which enhances elemental diffusion capability—a primary factor enabling solid-state reactions to occur and complete at the relatively low room temperature. Additionally, whether solid-state synthesis reactions occur during ball milling depends on the degree of energy increase in the system, while reaction completion is controlled by diffusion processes—namely, the degree of grain refinement and powder collision temperature. During high-energy ball milling, severe plastic deformation from powder collisions converts deformation energy into thermal energy, causing temperature rise in powders and promoting solid-state phase transformations and diffusion. Yang et al. modeled and estimated powder temperature rise and energy accumulation during ball milling, showing that temperature rise during powder collisions depends on collision velocity, angle, and powder material type; since powder cooling time is much shorter than ball flight time, powders do not accumulate energy and local melting does not occur. The temperature rise effect during ball milling promotes elemental diffusion and solid-state reactions. Therefore, during high-energy ball milling, Y_2O_3 particles are broken down into Y and O atoms, enabling them to undergo solid-state reactions with CLAM steel powder and completely dissolve into it, ultimately producing homogenized alloyed powder.

[Figure 4: see original paper] shows XRD spectra of CLAM steel powder with 0.3% Y_2O_3 after ball milling for different times. No Y_2O_3 diffraction peaks are observed in the XRD spectra because the Y_2O_3 content is low and partially broken down and dissolved into CLAM steel powder. Grain size and lattice distortion of CLAM steel powder after different milling times can be calculated from diffraction angles and half-width values of diffraction peaks.

Crystal diffraction must satisfy Bragg’ s equation:

$$2d\sin\theta = n\lambda$$

where d is interplanar spacing. The relationship between powder crystallite size D, diffraction angle , and half-width $B_1/_2$ can be expressed by the Scherrer formula:

$$B_{1/2} = \frac{K\lambda}{D\cos\theta}$$

where K is a constant (0.89) and λ is X-ray wavelength (0.15405 nm). During mechanical alloying, the mixed powder undergoes repeated cold welding-fragmentation and severe plastic deformation, promoting interdiffusion or solid-state phase transformations between powder particles and causing lattice distortion in micro-regions of powder particles, leading to changes in lattice constants. According to Bragg’ s equation, when lattice constants increase, diffraction angles decrease and diffraction peaks shift left; conversely, peaks shift right. After lattice distortion occurs, small deviations exist between incident and diffraction angles of X-rays, reducing diffraction intensity. Superposition of multiple diffraction peaks with deviations leads to peak broadening in XRD spectra. Diffraction peak broadening B caused by lattice distortion is:

$$B_\varepsilon = 4\varepsilon \cdot \tan\theta$$

Therefore, the half-width B expression considering both lattice distortion and size effects should be:

$$B = \frac{K\lambda}{D\cos\theta} + 4\varepsilon \cdot \tan\theta$$

where B, , and D can be read from XRD spectra. By plotting (Bcos)/λ versus sin /λ for each diffraction peak and fitting a straight line, the slope gives 4 and the intercept gives K/D. Table 3 lists peak positions, peak shifts, half-widths, crystallite sizes, and lattice strains of the (110)α-(Fe,Cr) diffraction peak for CLAM steel powder after different milling times.

XRD analysis and calculations show that after high-energy ball milling, the (110)α-(Fe,Cr) diffraction peaks all shift rightward. The standard

(110)α-(Fe,Cr) diffraction peak position is 44.484°. Calculating the offset of (110)α-(Fe,Cr) peaks after different milling times reveals that after 50 h of milling, the peak shows the maximum offset relative to the standard position, approximately 3.46‰. Additionally, with increasing milling time, the intensity of (110)α-(Fe,Cr) diffraction peaks gradually decreases while half-width increases. At 60 h of milling, the half-width reaches its maximum value of 0.786°, with lattice strain reaching 0.627%. After 10 h of milling, CLAM steel powder has a crystallite size of 127 nm and lattice strain of 0.545%; during 20-60 h of milling, crystallite size does not change significantly, only decreasing from 117 nm to 110 nm, but lattice strain reaches 0.582-0.684%, much higher than after 10 h. This indicates that during 20-60 h of milling, lattice distortion is the main cause of peak broadening. After high-energy ball milling, CLAM steel mixed powder undergoes severe plastic deformation, introducing numerous defects and large lattice distortion, achieving the highest degree of alloying with Y_2O_3 particles completely dissolved in CLAM steel powder particles, providing favorable conditions for generating fine Y_2O_3 particles during subsequent consolidation.

[Figure 5: see original paper] shows the distribution of alloying elements in large powder particles after 50 h of milling. After milling, Cr, Mn, and V elements are uniformly distributed in powder particles, while W and Ta contents are low. This is because most Ta element was burned off during CLAM steel atomization, resulting in very few bright spots in the mapping image; W element may have segregated during atomization due to its high density, leading to non-uniform distribution among particles. Additionally, after high-energy ball milling, Y_2O_3 particles no longer exist as oxides but are broken down, with Y atoms dissolving into the fresh surfaces of CLAM steel powder that undergo continuous cold welding and fragmentation, resulting in uniform Y distribution throughout CLAM steel powder particles. However, in larger CLAM steel powder particles (>20 µm), Y distribution is non-uniform, mainly concentrated within 5-8 µm from the surface, with lower Y content in particle cores. This indicates that the optimal CLAM steel powder size after milling is 10-16 µm, within which Y can be uniformly distributed throughout the powder particles, facilitating more dispersed Y_2O_3 particle distribution during subsequent consolidation. For the CLAM/Y_2O_3 system, CLAM steel powder particles are ductile while Y_2O_3 particles are brittle. Murty and Ranganathan proposed that in mechanical alloying of ductile/brittle systems, the deformation behaviors differ. During high-energy ball milling, ductile particles undergo severe plastic deformation, are flattened by grinding balls, and continuously cold-weld and fragment; brittle particles are broken down and embedded in severely deformed ductile particles, with the small size of fragmented brittle particles shortening diffusion distances. With extended milling time, ductile particle size gradually decreases and system temperature rises, promoting diffusion of metallic elements from brittle particles into ductile particles. Therefore, for the CLAM/Y_2O_3 system, Y_2O_3 is first broken down and encapsulated by CLAM steel during milling, and with temperature increase, Y atoms diffuse into CLAM steel powder particles, eventually leading to Y enrichment in CLAM steel powder surfaces and lower Y content in cores. To eliminate this Y enrichment, milling energy should be further increased to cause more severe plastic deformation of CLAM steel powder, forming flatter particles and improving dispersion of broken Y_2O_3 particles to achieve uniform Y distribution.

Based on the above discussion, longer ball milling times consume more energy, which is unfavorable for industrial production of ODS-CLAM steel. Additionally, severely plastically deformed powder particles after long-time milling exhibit improved alloying degree, significantly reduced particle size, and large lattice distortion. Considering these factors comprehensively, the optimal ball milling parameters are selected as: milling time 50 h, Ar atmosphere, ball-to-powder ratio 10:1, and 6 mm diameter hard steel balls as milling media.

2.2 High-Temperature Microstructural Stability of ODS-CLAM Steel

To investigate the high-temperature microstructural stability of ODS-CLAM steel, both CLAM and ODS-CLAM steels were solution-treated at 950–1200 °C for 30–60 min. Grain sizes before and after solution treatment were measured using the intercept method in metallographic analysis software SISCIAS 8.0. [Figure 6: see original paper] shows OM images of CLAM steel before and after solution treatment at 1200 °C. Before solution treatment, CLAM steel exhibits equiaxed grains with a size of 15.2 µm (Fig. 6a); after solution treatment at 1200 °C for 60 min, grain size increases to 197.0 µm (Fig. 6b). [Figure 7: see original paper] shows OM images of ODS-CLAM steel before and after solution treatment at various temperatures. To further improve mechanical properties, the HIP-consolidated ODS steel was hot-rolled, resulting in a fibrous microstructure with grains elongated along the rolling direction. Before solution treatment, ODS-CLAM steel has a grain size of 20.0 µm (Fig. 7a); after solution treatment at 950 °C for 30 min, grain size is 34.5 µm (Fig. 7b); at 1000 °C for 30 min, grain size is 49.8 µm (Fig. 7c); at 1200 °C for 60 min, grain size is 63.0 µm (Fig. 7d). Comparing grain sizes of CLAM and ODS-CLAM steels after solution treatment at 1200 °C for 60 min, CLAM steel grain size becomes 16.4 times its original size, while ODS-CLAM steel grain size becomes only 3.2 times its original size. This indicates that after solution treatment at 1200 °C for 60 min, ODS-CLAM steel exhibits significantly less grain coarsening than CLAM steel, demonstrating superior high-temperature microstructural stability.

[Figure 8: see original paper]a and b show TEM images of CLAM steel before and after solution treatment at 1200 °C. The as-heat-treated CLAM steel exhibits uniform lath sizes (200–300 nm) with fine $M_{23}C_6$ carbides and MX carbonitrides distributed along lath boundaries. [Figure 8: see original paper]c–f present SAED patterns and EDS spectra of $M_{23}C_6$ carbides and MX carbonitrides from Fig. 8a. SAED and EDS analysis identifies Fig. 8c as the [211] zone axis electron diffraction pattern of $M_{23}C_6$ with composition 0.4V-65.9Cr-1.7Mn-27.2Fe-4.8W (at%); Fig. 8d is the [111] zone axis pattern of TaC with composition 11.1Cr-79.4Fe-9.5Ta (at%). After solution treatment at 1200 °C for 60 min, CLAM steel martensitic laths widen to 400–500 nm due to the high temperature, and $M_{23}C_6$-type carbides and MX-type carbonitrides on lath boundaries dissolve completely, with Cr, Ta, and V alloying elements fully dissolved into the matrix (Fig. 8b). [Figure 9: see original paper] shows TEM images of ODS-CLAM steel before and after solution treatment at 1200 °C. The as-heat-treated ODS-CLAM steel contains $M_{23}C_6$-type carbides and MX-type carbonitrides (Fig. 9a). However, after solution treatment at 1200 °C for 60 min, martensitic laths remain fine at approximately 200 nm (Fig. 9b), $M_{23}C_6$-type carbides and MX-type carbonitrides have completely dissolved into the matrix, but nanoscale Y_2O_3 particles dispersed within laths and along lath boundaries remain clearly visible (Fig. 9c). This indicates that under high-temperature conditions, nanoscale Y_2O_3 particles can still provide dispersion strengthening in ODS-CLAM steel, ensuring good high-temperature mechanical properties. Furthermore, Ramar and Schäublin observed Y_2O_3 particle changes in ODS-Eurofer steel during in-situ heating, finding that carbides dissolved at 800 °C while Y_2O_3 particles remained undissolved; when temperature increased further to 1000 °C, Y_2O_3 particles remained stable in the matrix and continued to pin dislocations. Thus, our results are consistent with Ramar and Schäublin' s findings. In summary, after holding at 1200 °C for 60 min, ODS-CLAM steel shows no significant grain growth and stable Y_2O_3 particles in the matrix, demonstrating significantly better microstructural stability than CLAM steel.

Conclusions

1. The particle size (D) of CLAM steel powder with 0.3% Y_2O_3 addition gradually decreases with extended ball milling time (t) following the relationship: lgD = 1.648 -0.012t.
2. The optimal ball milling parameters for CLAM steel powder with 0.3% Y_2O_3 are: milling time 50 h, Ar atmosphere, ball-to-powder ratio 10:1, and 6 mm diameter hard steel balls as milling media.
3. To ensure uniform Y distribution after high-energy ball milling, CLAM steel powder particle size should be controlled at 10–16 µm. In CLAM steel powder particles larger than 20 µm, Y distribution is non-uniform, mainly concentrated within 5–8 µm from the surface, with lower Y content in particle cores.
4. Compared with CLAM steel, ODS-CLAM steel exhibits significantly less grain coarsening after solution treatment at 1200 °C for 60 min, demonstrating superior high-temperature microstructural stability.

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