Effect of explosive compaction on microstructure of ODS FeCrAl alloy fabricated by oxidation method

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Keywords: ODS FeCrAl alloy, explosive compaction, defects, Y-Al-O nanoparticles, prior powder boundary

Abstract
A fabrication process of 14Cr ODS FeCrAl alloy including direct oxidation treatment, explosive compaction and vacuum post-sintering was proposed herein. A thin oxide layer formed on the powder surface during a low-temperature oxidation treatment was crushed in the explosive compaction, resulting in the redistribution of oxides at prior powder boundary (PPB) and the subsequent formation of an exclusive iron oxide layer between the grains of compacted materials. A large number of defects (dislocations and grain boundaries) were also produced in the compacted particles. Through characterizing the evolution of oxide dispersoids during the course of fabrication, the contribution of explosive compaction to dislocations, nanoscale precipitates and mechanical properties was clarified. The results show that the iron oxide layer became the container for transporting oxygen and was dissolved during the post-sintering, which affected the composition of nanoscale precipitates. The high-density dislocations not only became the rapid diffusion paths of oxygen, aluminum and yttrium atom, but also became the preferential sites of Y-Al-O precipitation. With the manufacturing method, the more uniform distribution of oxide nanoparticles and the higher microhardness of ODS alloy were obtained.

1. Introduction

Oxide dispersion strengthened (ODS) FeCrAl alloys have drawn wide attention as a promising candidate structural materials for Generation IV nuclear fusion reactors due to their superior creep strength, excellent oxidation and irradiation resistance at elevated temperatures \[1\]. Its distinguished properties are caused by the homogeneous dispersion of fine nanoparticles mainly composed of Y-Al-O phase, which play an important role in acting as the pinning point for dislocation and grain movement \[2\]. ODS ferritic alloys are conventionally fabricated by mechanical alloying (MA) and subsequent consolidation. The dissociation of yttria particles and the introduction of oxygen are realized in the process of MA, which usually takes tens or even hundreds of hours \[3, 4\]. Hereafter, the canned powders are subjected to hot isostatic pressing (HIP) or hot extrusion (HE) at high temperatures and pressures, for the re-precipitation of nanoscale oxide particles in the consolidated material. The whole manufacturing process has been proven to be lengthy and costly. In addition, several drawbacks caused by long-term MA procedure, such as the inevitable contamination from grinding balls and jar, uncontrollable increases in oxygen or carbon, have been highlighted. To address these issues, various oxidation methods have been developed for avoiding MA procedure \[5–7\]. In the study of gas atomization reaction synthesis (GARS) and surface treatment of gas atomized powder followed by reactive synthesis (STARS), Rieken \textit{et al} \[8\] and Gil \textit{et al} \[9\] respectively used a small quantity of oxygen mixed in the atomization atmosphere to form a thin Cr-enriched metastable oxide layer on the as-atomized FeCr powder surface. These powders became the precursor powders for the generation of Y- and Ti-enriched oxide dispersoids in subsequent consolidation. Li \textit{et al} \[10\] proposed a process of vacuum heat
treatment followed by slow oxidation, and prior to HIP procedure a nanoscale yttrium oxide layer was formed at
the grain boundary and powder surface. Y-Al complex nanoscale oxides with a number density higher than
$1.8 \times 10^{21} \text{ m}^{-3}$ were obtained in the FeCrAl matrix by reasonable sintering. More recently, our study shows that
Y-Al complex oxides with a number density approaching $5.18 \times 10^{21} \text{ m}^{-3}$ can be identified in ODS FeCrAl alloy
by means of a simple direct oxidation method [11]. Nevertheless, the oxidation methods for preparing ODS alloys
are still questionable, e.g. poor number of precipitates, heterogeneous distribution of second phase particles.

During the oxidation process, a key step is to form metastable oxide layers or small oxide dispersoids on
particle surface and grain boundaries of yttrium-containing powder in a controllable oxygen environment.
These oxide layers and oxide dispersoids dissociate during subsequent consolidation, and then react with
yttrium or yttrium oxide through the available diffusion of oxygen along particle surface and grain boundaries,
thus producing complex oxide nanoparticles [11, 12]. Therefore, the increase in specific surface area and grain
boundary should be conducive to the precipitation of complex oxide nanoparticles. Additionally, the defects are
easily generated in the powder due to the kneading action in the process of MA, and these defects have been
proven to be the preferential nucleation sites of oxide nanoparticles during consolidation [13, 14]. However, this
is a challenging issue for the powder used in the oxidation methods, since the powder will not be damaged in the
absence of ball-milling procedure and there exist few defects. On the other hand, explosive compaction, as a
promising near-full-density powder metallurgy forming process, has been applied in the fabrication of new
materials with unique microstructure, such as superconductor [15] and high entropy alloy [16]. In the explosive
compaction process, the destructive force causes cracks on the powder surface and increases lattice defect as well
as dislocation substructure in powder. The accompanying high stress and high temperature reduce the porosity
and bond the powder particles, leading to the production of unexpected microstructures and properties [17]. To
date no studies have been reported on explosive compaction employed for the fabrication of ODS alloy.

In this paper, explosive compaction technique after oxidation treatment is introduced, and the fabrication of
14Cr ODS FeCrAl alloy by oxidation process followed by explosive compaction and sintering is described. The
evolution of oxide dispersoids in powder and ODS alloy is expounded. The effects of explosion on
microstructure and properties of powder and alloy are discussed.

2. Experimental procedure

The precursor material used in this study was 14Cr FeCrAl as-oxidized powder with particle size less than 53 $\mu$m.
The detailed procedure of oxidation is the same as that described in the [11]. The final oxygen content of
powders was 0.11 wt.%. About 3.5 kg of as-oxidized powder was packed in a container made of 304 stainless steel
(SS) with a diameter of 70 mm and a height of 240 mm. Two thick plates made of 304SS were put into the
container at intervals to improve the shock-compacted effect. After being pressed by a hydraulic press machine
for three times, the container (hereafter called tube) was sealed and degassed in vacuum at 400 °C for 4 h.

Figure 1 shows a schematic diagram of the selected explosive compaction set-up. The stainless steel tube
filled with powder was placed in the center of a PVC tube, and the space between metal tube and PVC tube was
packed with explosive material with uniform density. The mass ratio of explosive to powder was approximately 2.7. The detonator was inserted into the explosive material and connected to an electric detonator. The explosive was detonated from one end of the assembly. After compaction and demoulding, the compacted material is designated as OX-14SC, and its detailed composition is listed in Table 1. Post-sintering of the compacted material was carried out in vacuum at 800 °C, 1000 °C and 1200 °C, respectively, with heating rate of 10 °C min⁻¹ and holding time of 3 h, to promote the formation of oxide nanoparticles. The sintered alloys are called OX-14SC8, OX-14SC10 and OX-14SC12, respectively.

The morphology of as-oxidized powder was observed by field emission scanning electron microscope (FE-SEM) using a Zeiss Sigma 500 with an EDS analyzer. After manual grinding, polishing and chemical etching, the metallography of compacted specimen was examined using a Zeiss Axio Observer Z1 optical microscope. The etching solution was composed of 35 g FeCl₃, 100 ml hydrochloric acid and 40 ml deionized water. The nanometric features in compacted specimen and ODS alloy were characterized by TEM and HRTEM using Talos F200X at a voltage of 200 kV. The microhardness of ODS alloy was measured on a Micromet 5103 Vickers hardness tester using Vickers diamond pyramid indentation with a load of 500 g. The hardness value was obtained by the average of 10 consecutive indentations.

### Table 1. Chemical composition of the compacted specimen (wt.%).

| Compact Fe Cr Al Y Ti O N |
|--------------------------|
| OX-14SC Bal. 13.95 4.51 0.51 0.55 0.11 0.0023 |

3. Results

3.1. Morphology of powder

Figure 2 shows the surface morphology of 14Cr FeCrAl powder before and after oxidation treatment. The surface of as-atomized powder is clean; while an appreciable amount of oxide dispersoids are generated on the powder surface after oxidation. The particle size of oxide dispersoids near the grain boundary is slightly larger than that in the grain, as shown in figure 2(b). The contents of yttrium and aluminum in the large oxide dispersoids near the grain boundary are higher than those of the powder, indicating that yttrium and aluminum are enriched in the form of oxides at the grain boundaries after long-term low temperature oxidation treatment. This is consistent with the results of our previous study [11]. In addition, our previous research also shows that an iron oxide layer was formed on the surface of the powder, and this oxide layer has been considered as the main oxygen reservoir for precipitating oxide complex nanoparticles.

3.2. Microstructure of compacted specimen

Morphology and metallographic micrograph of OX–14SC specimen are shown in Figure 3. The shrinkage rate of 9% to 11% is obtained by the explosive compaction. The final shape of the compacted material is nearly cylindrical. No obvious macroscale defects such as circumferential or transverse cracks were found.
As shown in the region I (marked by the black character) of figure 3(b), the original microstructure of some powders remains in the compacted specimen, concomitant with prior particle boundary (PPB, marked with blue dashed line). According to the result of figure 2(b), the PPB should be composed of Al- and Y-enriched oxide dispersoids near the grain boundary and iron oxide film on the powder surface. Due to the inhomogeneous transfer of shock pressure and thermal energy from outside to inside, some welding microstructures derived from melting and rapid solidification between powder particles are observed in the region marked with II, which exhibits very fine grain structure. And some jet-like grains are observed in the dark region marked with III. These are the typical characteristics of explosive compacted powder [18]. Under the interaction of explosive force, the powder materials quickly flow into and fill the voids between powders, forming grain jets [19]. The adjacent particle marked with IV is subjected to an impact of the jet, so that the distorted grains are observed [20]. In the optical micrograph, no pores and voids were found between the powder particles, which indicate that the metallurgical bonding of FeCrAl powder has been formed during explosive compaction.

In order to explain the influence of explosive compaction on the as-oxidized powder, the microstructure between particles of the compacted specimen was also characterized by TEM, and the result is shown in figure 4. In the bright field image of TEM, the boundary width marked by red arrow is significantly larger than that marked by blue arrow. This is mainly because the boundary marked by blue arrow is an intrinsic grain boundary, while the boundary marked by red arrow is obtained by melting powder surface and subsequent welding [21, 22]. With the aid of XEDS analysis, the distribution results of element near the welding layer are shown in figure 4(b). It is found that aluminium and yttrium are concentrated in the welding layer, that is, the interparticle region (marked by red circle) and the outer surface of the powder. The width of aluminium and yttrium intermetallics is about 100 nm. A layer composed of iron and oxygen with a thickness of approximately 90 nm is also detected inside the powder surface. In this oxide layer the enrichment of aluminium, yttrium and
chromium cannot be identified. This suggests that an exclusive iron oxide layer has been formed at the particle interface after explosive compaction.

It is well-known that aluminum and yttrium have strong affinity for oxygen. Nevertheless, by investigating the grain boundary segregation of yttrium-doped polycrystalline titanium dioxide, Wang et al [23] found that driven by elastic strain energy, yttrium has segregated at grain boundaries and outer surfaces, and Y₂Ti₂O₇ precipitate has formed locally. Through analyzing the segregation phenomenon of Y in α-Al₂O₃, Wang et al [24] concluded that aluminum and yttrium can rapidly segregate from the oxides to the outer surface of powder and grain boundary at the high temperature. The authors speculate that the high stress and local high temperature caused by explosion promote the rapid segregation of aluminium and yttrium along the grain boundary to the particle outer surface, and then formed intermetallic compounds at vacuum environment. The remaining oxygen is combined with nearby iron to form an exclusive iron oxide layer. Therefore, the chemistry at PPBs in figure 2(b) has changed from the mixed alumina-, yttrium-enriched and iron oxides to an exclusive iron oxide layer after explosive compaction. In the previous study [11], it was only inferred that iron oxide is the oxygen container of oxide nanoparticles precipitation. However, the micro-environment between welding powders formed after explosion as shown in figure 4 provides direct evidence that iron oxide is the main oxygen reservoir for precipitating oxide complex nanoparticles during the following sintering. Compared with the result confirmed by Riken et al [8] and Li et al [10], the composition of metastable oxide layer is different. It indicates that the composition of intermediate oxide products in the oxidation method is controlled by the specific fabrication process.

Figure 5 shows the dislocation substructure of OX-14SC specimen. Appreciable amounts of dislocations are produced after explosive compaction. The phenomenon is obviously distinct from the as-oxidized powder prior to explosion and the ODS alloys fabricated by oxidation methods [8, 10, 11, 25]. Taking into account the role of dislocation in the conventional MA methods, it can be expected that the oxide precipitation would be promoted in the subsequent consolidation process.

As discovered by Vorozhtsov et al [26], the metastable second aluminium phase was formed in the aluminium-based composites after shock wave compaction. The oxide nanoparticles in the OX-14SC specimen were also characterized by TEM. Figure 6 shows the distribution of second phase particles and the corresponding element distribution result of a nanoparticle in the matrix. The dispersed particles are very fine, but the numbers are very small. Most particles (marked by blue arrows) were observed to be aligned along the grain boundaries. The element analysis of particle with a size of around 10 nm (marked with red circle) in figure 6(a) reveals that the precipitate is enriched in yttrium, aluminum and oxygen, indicating the formation of Y-Al complex oxides. During the explosion, the total amount of thermal energy transferred to the material in this region is large enough, which exceeds the activation energy required for the complex reaction between aluminum and yttrium oxide. Therefore, Y-Al complex oxides with small sizes are formed. However, since the thermal energy generated by shock wave propagation is localized and instantaneous [18, 27], high-density oxide nanoparticles cannot be produced in the compacted specimen.

3.3. Nanometric features of as-sintered alloy
To obtain high-density oxide nanoparticles, the post-sintering of compacted specimen at different temperatures was carried out. Figure 7 shows the nanometric features in the ODS alloy sintered at 800 °C, 1000 °C and 1200 °C, respectively.
Figure 6. (a) Distribution of nanoparticles in the compacted specimen, (b) corresponding elemental mapping result for a nanoparticle in (a). The blue dash lines represent the grain boundaries in the compact.

Figure 7. TEM images of nanoparticles in ODS alloys OX-14SC8, OX-14SC10 and OX-14SC12: (a) (e) in the grain, (b) (f) near the grain boundary.
In the alloy OX-14SC8, a number of particles less than 10 nm are finely dispersed in the grain. The average size of nanoparticles is 2.65 nm, and the number density is 1.74 \times 10^{22} \text{ m}^{-3} (calculated by the thickness of 50 nm). In addition, some coarse particles are precipitated along the grain boundary, while the very small particles are dispersed away from the grain boundary. These particles exhibit a wide range of size distribution, ranging from very small spheres of less than 3 nm to oval-shaped with a maximum size of 130 nm. The coarse particle with an irregular morphology in figure 7(b) appears to be aggregations comprised of small precipitates. The chemical composition of these coarse particles in figure 7(b) was determined by EDS, and the result is listed in table 2. The particles I, IV and V (marked with the yellow character) in OX-14SC8 are detected to contain near amounts of iron and oxygen, and negligible amounts of aluminium, yttrium and titanium. Based on the element analysis in figure 4, it is inferred that these coarse particles along the grain boundary should be the residue of iron oxide layer. During the post-sintering process, the high temperature treatments drive the particles to further bond, thereby attaining a fully dense microstructure [28]. With the diffusion of oxygen from the iron oxide to yttrium and aluminium, the iron oxide layer gradually dissolves. However, the thermal energy at 800 °C is not sufficient to dissolve all the oxides. Thus, iron oxides are retained and distributed at the PPB. At the same time, the relatively small particles marked with II and III are determined to contain a considerable amount of yttrium-aluminium complex oxides and yttria particles are formed. In OX-14SC8 specimen, no \text{Al}_2\text{O}_3 particles were found by TEM.

As shown in figure 7(c), large numbers of fine spherical nanoparticles are uniformly distributed in the grain of alloy OX-14SC10. The particle size is mainly concentrated in 5 nm, and the number density of nanoparticles is approaching 5.6 \times 10^{22} \text{ m}^{-3} (calculated by the thickness of 50 nm). It is close to the result of Wu et al [29] who prepared ODS FeCrAl alloy with the same chemical composition by conventional MA method. Also, the coarse and irregular particles at the grain boundary are observed in figure 7(d). The EDS result listed in table 3 shows that the irregular particles (marked with the yellow character) are mainly composed of Y-enriched oxide and Y-Al complex oxide. With the decrease in size, the aluminum content of particle increases. It is worth noting that iron oxide particles were not detected near the grain boundary. The result provides evidence that the PPB oxides have been completely dissolved at 1000 °C, concomitant with the available delivery of oxygen from PPBs to yttrium and aluminium. In comparison with the required sintering parameter (1300 °C) of ODS-FeCr alloy fabricated by GARS (Gas atomization reaction synthesis) [8] and STARS (Surface Treatment of gas Atomized powder followed by Reactive Synthesis) [25], the consolidation temperature at which PPBs are completely dissolved is considerably reduced. The authors speculate that this reduction mainly originates from the microstructure changes caused by explosive compaction, such as the redistribution of PPB oxide and the increase in defects. Moreover, some dislocations have been observed in the alloy OX-14SC10. Figure 8 shows the configuration between dislocations and oxide nanoparticles. The high-density oxide particles are entangled with the high-density dislocations. This seems to give evidence that the dislocations are preferential nucleation sites for the precipitation of oxides nanoparticles.

| Table 2. Composition of particles at the grain boundary in figure 7(b) (wt.%). |
|-----------------|---|---|---|---|---|---|
| Element  | Fe  | Cr  | Al  | Y  | Ti | O  |
| I       | 80.99 | 11.73 | 0.34 | 0.18 | 0 | 6.76 |
| II      | 14.39 | 4.63 | 28.47 | 20.41 | 0.97 | 30.83 |
| III     | 26.79 | 8.37 | 0.1 | 49.98 | 0.10 | 14.66 |
| IV      | 73.73 | 12.74 | 2.63 | 1.06 | 0 | 9.84 |
| V       | 78.56 | 11.53 | 0.21 | 0.13 | 0 | 9.57 |

| Table 3. Composition of particles at the grain boundary in figure 7(d) (wt.%). |
|-----------------|---|---|---|---|---|---|
| Element  | Fe  | Cr  | Al  | Y  | Ti | O  |
| I       | 25.73 | 8.43 | 0.1 | 49.08 | 0.49 | 16.17 |
| II      | 23.51 | 7.15 | 0.47 | 55 | 0.27 | 13.60 |
| III     | 23.73 | 7.21 | 23.83 | 16.63 | 0 | 28.60 |

Figure 7(e) and (f) shows the morphology and distribution of oxide particles in the grain and at the grain boundary of OX-14SC12. In the grain some coarse particles with a diameter of 50–200 nm are also found, in addition to a large number of small particles. According to the statistics of five TEM fields of view, the nanoparticles less than 15 nm account for about 90% of the total precipitates. The average particle size is 8.23 nm and the number density is 2.6 \times 10^{22} \text{ m}^{-3}. Near the grain boundaries the coarser and more irregular precipitates are observed in figure 7(f). Similar with the result of OX-14SC10, EDS analysis in table 4 shows that...
the coarse particles marked with I, II and III are predominantly composed of yttrium-enriched oxide and Y-Al complex oxide, and do not contain aluminium-enriched oxide.

To obtain structural information of the nanoscale oxides, the crystallographic features of particles with different sizes labeled as 1, 2 and 3 in figure 7 are determined by Selected Area Electron Diffraction (SAED) and Fast Fourier transformation (FFT) patterns, respectively. The diffraction pattern of the largest particle labeled as 1 (∼130 nm) is shown in figure 9(a), which is matched with YAlO3 phase with an orthorhombic structure (PDF: #33–0041). The result shows that the coarse particle within the grain is composed of YAlO3 phase. Compared to the particles with a similar size marked as I and II at the grain boundary in figure 7(f), the chemical composition is obviously different. The HRTEM image of particle 2 and corresponding diffraction pattern from FFT are shown in figure 9(b). The measured values of inter-planar distances (d) and angles (α) from the reciprocal space are consistent with those of YAlO3 phase with an orthorhombic structure (PDF: #33–0041), and they are listed in table 5 (the electron beam is paralleled to [001] zone axis of this particle). The HRTEM image of fine particle 3 and corresponding diffraction pattern from FFT are shown in figure 9(c) and (d). The inter-planar spacing measured from the reciprocal space (figure 9(d)) and the corresponding theoretical crystal inter-planar distances (d) of this oxide are listed in table 6. Within the error allowance, the particle is also identified to be YAO3 phase with an orthorhombic structure (PDF: #33–0041) (the electron beam is paralleled to [1 1 0] zone axis of this particle). Therefore, it can be concluded that the nanoscale oxide dispersoids within the grains are mainly composed of YAO3 phase. According to the crystal structure analysis of more than 40 particles, bcc-Y3Al5O12 (YAG), monoclinic-Al2Y4O9 (YAM), hexagonal-YAO3 (YAH) and Al2O3 are not distinguished.

### Table 4. Composition of coarse particles at the grain boundary in figure 7(f) (wt.%).

| Element | Fe  | Cr  | Al  | Y   | Ti  | O   |
|---------|-----|-----|-----|-----|-----|-----|
| I       | 22.53 | 7.68 | 0.3 | 43.56 | 0.31 | 25.62 |
| II      | 24.36 | 8.21 | 0.51 | 48.54 | 0.16 | 18.22 |
| III     | 21.59 | 7.64 | 25.74 | 18.67 | 0.12 | 26.24 |

3.4. Hardness of as-sintered alloy

After post-sintering at different temperatures, the microhardness of OX-14SC8, OX-14SC10 and OX-14SC12 alloys is 291.2 ± 11.1 Hv0.5, 325.7 ± 9.6 Hv0.5 and 297.6 ± 10.5 Hv0.5, respectively. With the increase of sintering temperature, the hardness increases first and then decreases, which is related to the number and distribution of oxide nanoparticles in these alloys [30]. The hardness value of OX-14SC12 is even higher than that (137 ± 3 Hv0.5) of ODS FeCr alloy fabricated by GARS route followed by heat treatment at 1200 °C [8]. Furthermore, compared with the result (275.2 ± 10 Hv0.5) of ODS FeCrAl alloy with the same composition fabricated by the same oxidation method but without the explosive compaction [11], the hardness of the three alloys is significantly improved. Actually, the hardness of the as-sintered OX-14SC10 alloy is even higher than
that of the as-HIPed ODS specimen (291–304 Hv<sub>0.5</sub>) manufactured by MA process [31]. It suggests that a certain amount of strain hardening exists in these as-sintered alloys. It also indicates that ODS FeCrAl alloy has good compactness, and there are a large number of oxide nanoparticles in the matrix, which is promoted by the introduction of explosive compaction.

**Table 5.** Inter-planar spacing (d) and angles (α) of particle 2 in figure 7(e) and the possible indexing.

|        | d<sub>1</sub>(010) | d<sub>2</sub>(110) | d<sub>3</sub>(100) | α<sub>12</sub> | α<sub>23</sub> |
|--------|-------------------|-------------------|-------------------|-------------|-------------|
|Measured| 7.367             | 4.383             | 5.392             | 54.73       | 90.19       |
|YAlO<sub>3</sub>| 7.370             | 4.318             | 5.328             | 54.13       | 90          |

**Table 6.** Inter-planar spacing (d) and angles (α) of particle 3 in figure 7(e) and the possible indexing.

|        | d<sub>1</sub>(11 1̅) | d<sub>2</sub>(220) | d<sub>3</sub>(111) | α<sub>12</sub> | α<sub>23</sub> |
|--------|----------------------|-------------------|-------------------|-------------|-------------|
|Measured| 3.296                | 2.133             | 3.294             | 39.56       | 39.48       |
|YAlO<sub>3</sub>| 3.316                | 2.159             | 3.316             | 39.81       | 39.81       |
Through analyzing the distribution of oxide nanoparticles in the matrix and measuring the hardness of ODS alloy, it is considered that 1000 °C is the most appropriate post-sintering temperature in this study. It is lower than the consolidation temperature required by the conventional MA process.

4. Discussion

As mentioned above, due to the most uniform distribution of oxide nanoparticles and the highest microhardness in OX-14SC10 alloy, the post-sintering temperature of 1000 °C is optimal for ODS FeCrAl alloy fabricated by oxidation and subsequent explosive compaction. It is commonly accepted that the oxide film on the powder surface can hinder the diffusion of atoms and affect the consolidation of the powder [32]. Pazos et al [6] and Gil et al [9] solidified the oxidized powder with HIP at 1220 °C–1300 °C. Through characterizing the nanoscale features in the ODS FeCrAl alloy after hot isostatic pressing at 900 °C, 1220 °C and 1300 °C, Ordás et al [12] concluded that the low temperature (900 °C) was not conducive to obtaining good microstructure, and the prior particle boundaries disappeared completely until the temperature rose to 1270 °C. In comparison with oxidation method without explosive compaction, the temperature required for the disappearance of the PPBs and the highest number density of oxide nanoparticles is markedly reduced. The most reasonable explanation is that the microstructure of interior and surface of as-oxidized powder has been changed by the explosive compaction.

Figure 10 shows the evolution of oxide dispersoids and the precipitation process of nanoparticles in the whole manufacturing process. As shown in figure 10(a), the oxide dispersoids enriched in aluminium and yttrium together with an oxide layer composed of iron oxide are formed on the powder surface after a simple oxidation treatment. Meanwhile, due to the strong diffusivity and oxygen affinity, the aluminium- and yttrium-enriched oxide dispersoids should also be distributed along the grain boundaries. During the explosion process, the high pressure caused by the shock wave enables the powder surface to be broken, the oxide layer to be fractured, and the defects (dislocations and grain boundaries) to occur. The fragmentation of particle surface and oxide layer shortens the diffusion route of element. At the same time, the shock wave propagates through the as-oxidized powder surface. The segregation of aluminium and yttrium to the grain boundary and powder surface is driven by the instantaneous high temperature. Because of the rapid cooling, a welding layer composed of aluminum and yttrium intermetallic compound is produced under vacuum condition. Thus, two kinds of layers are formed between powder particles or grain boundaries: the outer layer is Al-Y intermetallics and the inner layer is iron oxide. Through the inward diffusion of oxygen from the oxide layer and the diffusion of aluminium and yttrium along the defects, the exclusive iron oxide layer becomes the oxygen reservoir to form complex oxide nanoparticles in the subsequent consolidation. On the other hand, the increased dislocations and grain boundaries provide a fast way for atoms to diffuse, and enhance the opportunity of combining each
element. At the same time, these defects become the nucleation sites of oxide nanoparticles. The above synergistic effects result in the relatively rapid elimination of the PPB and the more uniform precipitation of complex oxide nanoparticles in the matrix.

The coarsening of oxide particles at the grain boundary was observed in all ODS alloys. Possible reasons are as follows: first, the residue of PPB oxides grows and coalesces. The sintering at different temperatures is performed to dissolve the oxides at PPBs, facilitate the diffusion of oxygen in ferrite grains and react with yttrium and aluminium in the matrix. According to the thermodynamic stability of Fe-based ODS alloy calculated by Chinnappan [33], Y-Al-O and Y_2O_3 particles have the almost identical negative formation enthalpies, and they can be formed simultaneously by slight change of microenvironment in the production process. In this investigation, yttrium near the iron oxide layer (as shown in Figure 4) can easily react with oxygen to become yttrium oxide due to its strong oxygen affinity, and then yttrium oxide particles coarsen during heating. This is why yttrium-aluminium complex and yttrium oxide particles are always identified at the grain boundaries simultaneously (seen tables 2–4). Since the iron oxide layer at PPB is partially dissociated at 800 °C, a coarsening of iron oxide particles near the grain boundary is found. Second, atomic diffusion occurs between the nanoscale particles at high temperature. Diffusion of atoms in a crystal lattice is a thermal activation process which can be strongly accelerated by defects. Using in situ TEM, Legros et al [34] observed that via a single dislocation line a Si atom diffused from smaller to larger precipitate on the Al thin film, until the smaller precipitate disappeared. Love [35] also confirmed that the diffusion rate of atoms along the dislocations and boundaries is several orders of magnitude faster than that of bulk diffusion. In the present investigation, the defects of the compacted powder increase sharply due to explosion. And then the increase of dislocations within the grains and near the grain boundaries increases the chance of combining each element and precipitating complex oxide nanoparticles. However, as Voorhees [36] and Simonsen et al [37] proved in the Ostwald ripening mechanism, the different excess surface energy of each precipitate drives atoms diffuse from small particles to large particles through dislocation lines and grain boundaries, resulting in the change in morphology and size of precipitates. Therefore, at high temperature, some coarse particles can be found within the grain and at the grain boundaries. More theoretical works on the coarsening of some nanoparticles during the oxidation fabrication will be covered in later research.

5. Conclusion

In this study, an explosive compaction technique was introduced into the direct oxidation method to fabricate 14Cr ODS FeCrAl alloy. The effect of explosion on microstructure and microhardness of alloy were explored. Results are summarized as follows:

(1) The deformation of as-oxidized particles, the fragmentation of powder surfaces, the welding of interparticles and the redistribution of prior particle boundaries take place during the explosive compaction. There are two layers between particles, namely, the outer layer is an intermetallic compound of yttrium and aluminum, and the inner layer is an exclusive iron oxide.

(2) Explosive compaction can also produce high density defects. In the following sintering process, the defects shorten the diffusion path of Y, Al and O atoms, and become the preferential nucleation sites of Y-Al-O nanoparticles, leading to an increase in nanoscale particles.

(3) The post-sintering at vacuum promotes the uniform distribution of Y-enriched nanoparticles in the matrix. The optimal sintering temperature is obtained to be 1000 °C.

(4) Due to the explosive compaction, the temperature required for complete dissolution of PPB oxides is reduced, and the hardness of ODS alloy is improved.

(5) In the 14Cr ODS FeCrAl alloy produced by this method, the nanoparticles within the grains are composed of YAlO_3 phase, and Y_2O_3 and YAlO_3 particles are distributed along the grain boundaries.

Acknowledgments

The authors acknowledge the support of this work by the National Defense Science and Technology Industry Nuclear Material Technology Innovation Center Project (No:ICNM-2020-ZH-17).
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