Microstructure and Mechanical Properties of Nano-Y_2O_3 Dispersed Ferritic Alloys Synthesized by Mechanical Alloying and Consolidated by Hydrostatic Extrusion

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Abstract:

The present study reports synthesis of 1.0 wt % nano-Y_2O_3 dispersed high strength ferritic alloys with nominal compositions of 83.0Fe-13.5Cr-2.0Al-0.5Ti (alloy A), 79.0Fe-17.5Cr-2.0Al-0.5Ti (alloy B), 75.0Fe-21.5Cr-2.0Al-0.5Ti (alloy C) and 71.0Fe-25.5Cr-2.0Al-0.5Ti (alloy D) (all in wt %) by mechanical alloying using planetary ball mill followed by consolidation of alloyed powders by hydrostatic extrusion at 1000°C and 550 MPa pressure with a strain rate ~ 10^{-1} s^{-1}. The products of mechanical alloying and extrusion have been characterized by X-ray diffraction, scanning and transmission electron microscopy, energy dispersive spectroscopy and image analysis. Mechanical properties in terms of hardness, compressive strength, yield strength and Young's modulus have been determined using nano-indenter and universal testing machine. The present ferritic alloys record significantly high levels of compressive strength (850-2226 MPa) and yield strength (525-1505 MPa), Young's modulus (240-265 GPa) and hardness (14.7-17.8 GPa) with an impressive true strain (5.0-22.5 %). This extraordinary strength level measures up to 1.5 times greater strength, albeit with a lower density (~ 7.4 Mg/m^3) than that of (< 1200 MPa) standard oxide dispersion strengthened ferritic alloys. Furthermore, the extent of plastic strain before failure in the present routine surpasses all previous attempts of identical synthesis but different consolidation routes for the same set of ferritic alloys. In general strength is higher along transverse than longitudinal direction of extrusion. It is conclude that uniform dispersion of nanometric (20-30 nm) Y_2O_3 (ex-situ) or $Y_2Ti_2O_7$ (in-situ) in high volume fraction along boundaries and within the grains

of high-Cr ferritic matrix is responsible for this unique combination of high strength and ductility in the present alloys developed by powder metallurgy route.

Keywords: Mechanical alloying, hydrostatic extrusion, dispersion hardening, ferritic alloy, microstructure, mechanical properties

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1. Introduction

Oxide dispersion strengthened (ODS) ferritic or ferritic/martensitic steels are considered most suitable for structural components exposed to elevated temperature in super thermal plants or nuclear reactors due to their ability to retain mechanical strength and resist oxidation/corrosion under extreme conditions of temperature and pressure [1-4]. Among usual alloying elements (substitutional), chromium provides stability of ferritic structure by solid solution hardening and induces oxidation/corrosion resistance by forming adherent oxide scale on the surface [5]. Similarly, small percentage of aluminum and titanium enhances corrosion and oxidation resistance [6]. Finally, dispersion of ultra fine yttria or similar rare earth oxide is useful to prevent grain boundary sliding and creep, fatigue and radiation damage at elevated temperature. Most of the ODS alloys use (9-14 wt %) Cr, (2-4 wt %) Al, (0.3-0.5 wt %) Ti and about (0.3-0.5 wt %) Y_2O_3. Attempts to explore development of ODS alloy with greater amount of these alloying elements have not been successful. Production of nano-oxide dispersed ferritic alloys in bulk quantity from appropriate elemental blend usually involves powder metallurgical route like mechanical alloying due to the wide difference in physical properties (melting point, density, solubility and diffusivity) of the concerned components (elements and compounds). The alloyed powders are subsequently compacted using hot extrusion [1,5], cold compaction and pressure-less sintering [6], high pressure sintering [7,8], equi-

channel angular pressing [9,10], laser sintering [11], pulse plasma sintering [12-14] and hot isostatic pressing [15-17]. Among all the methods hot isostatic pressing yields a more isotropic microstructure but a lower ductility and fracture toughness as compared to other methods of compaction and sintering. In this regard, recent studies suggest that hydrostatic extrusion [18-20] can yield significantly isotropic and enhanced mechanical properties with improved density of products consolidated from oxide dispersed feeritic steel from mechanically alloyed powder mass [20].

Unlike indirect or direct extrusion, hydrostatic extrusion which is an improvisation of classical hot extrusion method, utilizes a high pressure fluid instead of a solid plunger to apply large compressive pressure on the billet from all sides and forces it to yield through a die. Usually, hydrostatic instead of uniaxial pressure induces better formability and results in increased shear stresses near the die entrance, reduced adiabatic heating during the process and better surface finish of the product [21- 23]. However, a detailed investigation is warranted to establish the correlation between microstructure and mechanical properties on one hand, and process parameters (pressure, temperature) on the other hand, particularly for ODS alloys with high alloy content (> 12wt.% Cr) synthesized by mechanical alloying and consolidated by hydrostatic extrusion. It may be pointed out that Karak et al. [24-27] have recently investigated scope of consolidation of the same set of alloys synthesized by identical mechanical alloying routine through pulse plasma sintering[24], hot isostaic pressing [25, 26] and high pressure sintering [27] to obtain superior combination mechanical properties and ductility/deformability. Despite high strength, these attempts could not achieve compressive ductility over 10 %.

In the present study, bulk nano-oxide dispersed ferritic alloys with high amount of Cr ranging from 12.5 -25.5 wt% and higher amount (1.0 wt %) of Y_2O_3 has been developed by mechanical alloying and subsequently high speed hydrostatic extrusion. Following this mechano-chemical synthesis and extrusion, the extruded coupons have been characterized by X-ray diffraction, scanning and transmission electron microscopy, energy dispersive spectroscopy and image analysis. Mechanical properties comprising hardness, compressive strength, yield strength, Young's modulus have been determined using nano-indenter and universal testing machine.

2. Experimental procedure

Appropriate amounts of pure Fe, Cr, Al, Ti and Y_2O_3 powders (≥ 99.5 wt % purity and 30-80 μm size) with initial blend compositions of 83.0Fe-13.5Cr-2.0Al-0.5Ti (alloy A), 79.0Fe-17.5Cr-2.0Al-0.5Ti (alloy B), 75.0Fe-21.5Cr-2.0Al-0.5Ti (alloy C), and 71.0Fe-25.5Cr-2.0Al-0.5Ti (alloy D) (all in wt %) each with 1.0 wt % nano-Y_2O_3 addition/dispersion were subjected to mechanical alloying in Retsch PM 400 high-energy planetary ball mill with 10:1 ball to powder mass ratio in stainless steel container with 10 mm diameter stainless steel balls at room temperature. Milling was carried out in wet (toluene) medium to prevent cold welding and agglomeration of the powders and to minimize oxidation during milling. The identity and sequence of the phase evolution at different stages of mechanical alloying were determined by X-ray diffraction (XRD) using Co-K_α (0.707 nm) radiation and scanning (SEM) and transmission electron microscopy (TEM). The grain size and grain distribution measurement were conducted by using careful image analysis.

The powder compacts were encapsulated/canned in low carbon steel containers of 50 mm diameter and 250 mm length which had a chemical composition (in wt%) of 0.35 C, 0.25 Si, 0.65 Mn, 0.2 Cr, 0.035 S, 0.03 P,0.2 Cu, 0.02 Ni and balance Fe. The containers with the specimens were heated to the deformation temperature (1000 °C) in a furnace in air, and the specimens were then placed into the working chamber of a hydrostatic extruder (Fig. 1a) [18, 19]. The canned billets were extruded using a conical die with 30° convergence angle at a reduction ratio of 25:1 (reduction ratio R = 4). Following hot extrusion, the billets were cooled to room temperature in air. The time lag before the onset of extrusion was 15–20 s. The extrusion temperatures were chosen after optimization of sintering temperature of the current alloys. The deformation was carried out in a horizontal hydrostatic extruder operating at pressures of up to 1.5 GPa. Castor oil was used as the working fluid. The strain rate during extrusion was about 40 mm/s (~ 10^{-1} s^{-1}).

Samples after hydrostatic extrusion develop typical cylindrical shape with a length of about 50 mm and diameter of 10 mm (as shown in Fig. 1b). Just like during mechanical alloying samples at different stages of extrusion were subjected to careful routines XRD, SEM and TEM analysis to determine phase identity, volume fraction and crystallite size of the sintered billets at various sections. The density was determined by using a helium pycnometer (AccuPyc 1330) [28]. The micro-composition of the different phases (spot, line or area profile) of the samples was determined by an EDS analysis along with microstructural studies using SEM and TEM.

Mechanical properties in terms of hardness and Young's modulus were determined from nano-indentation test on selected sintered samples using standard nano-

indentation experiment (TriboIndenter with MultiRange NanoProbe, Hysitron) with a Berkovitz indenter at 200 mN load. Each hardness value was measured from an average of 25 point measurements for nano-indentation and repeated 3-5 times at equivalent locations to ensure precision. Specimens with square cross-section and approximate dimensions of 3 mm × 3 mm × 6 mm were cut from the sintered samples for compression tests at room temperature tests in a 10 kN universal testing machine with tungsten carbide anvils operated at a strain rate of 1.0×10^{-3} s^{-1}. The load and displacement were measured using a quartz load cell with an accuracy of ± 1.0 N and an extensometer with an accuracy of ± 1.0 μm, respectively. During the compression test, the commencement of micro-cracking was monitored by acoustic emission method [29]. The fracture surfaces after compression tests were studied using SEM.

3. Results and Discussion

3.1 Phase evolution during milled and after hydrostatic extrusion condition

As already stated, the current alloys were synthesized from elemental powder blends of predetermined composition by mechanical alloying in high energy planetary ball mill for cumulative period of up to 40 h. Figs. 2(a-d)) show the XRD profiles these powder blends following mechanical alloying for appropriate durations (1-40 h) and consolidation by hydrostatic extrusion at 1000 °C using uniform 550 MPa pressure with a strain rate of ~ 10^{-1} s^{-1} for (a) alloy A, (b) alloy B, (c) alloy C and (d) alloy D, respectively.

It is apparent that the final milled product (after 40 h) in each case is a single phase body centre cubic (BCC) solid solution indicating that Cr, Al and Ti completely

dissolve in Fe in course of high-energy ball milling for up to 40 h. Furthermore, it appears that BCC-Fe(Cr) phase is the predominant constituent in all alloys along with intermetallic phases like $Fe_{11}TiY$ and $Al_{9.22}Cr_{2.78}Y$ and mixed oxide phase $Y_2Ti_2O_7$ after the hydro-extrusion of the alloys at 1000°C. The sequence of phase evolution in all the four alloys during synthesis by mechanical alloying but consolidated by other supplementary routes are precisely identical as that recently reported elsewhere by Karak et al. [24-27]. However, the alloyed powders used for the present study were synthesized as a separate set of powder mass exclusively for this study.

3.2 Microstructural analysis and grain morphology after hydrostatic extrusion

Figs. 3 (a-d) show the SEM images of the consolidated product prepared by hydrostatic extrusion and taken from the transverse direction of extrusion for alloy A, alloy B, alloy C and alloy D, respectively. It is found that the degree of grain refinement obtained from hydrostatic extrusion progressively increased with increase in Cr content and is the maximum in alloy D (Fig. 4d). It is interesting to note that besides grain refinement, the highest Cr containing alloy D registers marked and identical degree of isotropy (nearly equi-axied grain) both along transverse and longitudinal direction of extrusion compared to that say, the least Cr containing alloy (alloy A as in Fig. 3a). Higher amount of Cr in solid solution enhances the elastic modulus and strain hardening, causing dynamic recryatallization during hydrostatic extrusion at 1000°C, as it is higher than the projected recrystilyzation temperature of the present alloys. The effect of Cr content on such microstructural refinement and isotropy in nano-Y_2O_3 dispersed ferritic alloys has not been investigated earlier, except by Matijasevic et al. [30], who have recently reported similar effect of Cr concentration on microstructural changes in binary

Fe-Cr and not multicomponent Fe-Cr-Al-Ti alloys. It may be noted that the crystallite/particle size of the alloy powder during mechanical alloying of alloy D is the finest among all the four alloys. On the other hands, the elongated morphology of grains along extrusion direction in alloy A (Fig. 4e) suggests that alloy A may possess the highest ductility among all the four alloys.

Microstructural evolution during hydrostatic extrusion is strongly depended on density of individual alloy, strain rate (~ 10^{-1} s^{-1}) and extent of plastic deformation. The higher the Cr content and the higher the strain rate (~ 10^{-1} s^{-1}), led to the greater the density of defects (particularly line defects or dislocations) in the extruded product leading to higher hardness and compressive strength. In situation like hydrostatic extrusion, the process is largely adiabatic in nature in localized region. The heat is generated due to high strain rate during plastic deformation by extrusion causing adiabatic rise in temperature, which can be estimated by the following equation (assuming all plastic work is transformed to heat) [20]:

$$Q_h = C_p \rho \Delta T, \quad (1)$$

where Q_h = amount of heat per unit volume (Q_h is equal to the value of extrusion pressure applied for extrusion), C_p is the specific heat, ρ is the density and ΔT is the temperature rise. ΔT varies for a given extrusion process due to variation in the extrusion pressure and composition of the individual alloy which may influence the cross-section reduction ratio and hardening behavior of the alloy. The temperature rise favors defect reorganization and new grain formation necessary for nucleation of recrystalized grains during extrusion. Among all the present alloys, alloy D exhibits highest theoretical density, hence experiences the least temperature rise for a given Q_h and undergoes the

maximum extent of grain refinement. On the other hand, alloy A possesses the lowest density, hence experiences the highest temperature rise and grain growth during extrusion as compared to the other alloys.

3.3 Grain size measurement and distribution

Figs. 4 (a-d) show the grain size distribution measured from the microstructures for all the current alloys. It is evident that the ultra fine grains were produced during hydrostatic extrusion process. A similar kind of results has been reported by Lewandowska et al. [20] for Eurofer 97 steel obtained by hydrostatic extrusion. A critical comparison of microstructure and grain size distribution confirms that the grain size distribution is bimodal or multi modal in nature and varies in the range 0.3 to 2.5 μm. As already stated, the grain size is the finest in alloy D and coarsest in alloy A. Our earlier studies also corroborate that alloy D exhibits the finest grain/crystallite size after mechanical alloying for alloy D as compared to other alloys [24-27], which can be attributed to higher Cr content in alloy D.

3.4 TEM analysis

Fig. 5 (a-b) shows the bright field TEM image of hydrostatically extruded alloy A revealing the presence of nanometric dispersion of ex-situ (Y_2O_3) or in-situ ($Y_2Ti_2O_7$) oxide particles and precipitated intermetallic ($Fe_{11}TiY$ and $Al_{9.22}Cr_{2.78}Y$) phases located along the grain boundary or at grain boundary triple points. It is interesting to note that the microstructure consists of both smaller and larger size matrix grains (BCC) besides the nanometric dispersoids/precipitates. Furthermore, the matrix grains are polygonal and fully recrystalized suggesting that hydrostatic extrusion at 1000°C allows complete dynamic recrystalization during this hot deformation process. Fig. 6c shows the

microstructure and morphology of alloy A in longitudinal direction after extrusion at 1000°C. It is apparent that some grains are elongated and the dispersiods (Y_2O_3 or $Y_2Ti_2O_7$) and precipitated intermetallic ($Fe_{11}TiY$ and $Al_{9.22}Cr_{2.78}Y$) particles are situated both along grain boundary and within grain body.

3.5 Density

Fig. 6 shows the variation of bulk density and porosity of the extruded samples, measured by pycnometer, as a function of Cr content (wt. %) for the four different alloys. As Cr content increases the density increases and porosity decreases in all the present alloys. Accordingly, alloy D shows the maximum density and minimum porosity and alloy A records the reverse trend. It may noted that the powder density (40 h milled sample) was maximum for alloy D as compared to that of other three alloys (alloy A, alloy B and alloy C), as recently reported by Karak et al. [24-27].

3.6 Evaluation of mechanical properties in both extrusion directions

Fig. 7 shows the variation of the Young's modulus and hardness of the hydrostatically extruded products of all the four alloys as a function of Cr content. It is apparent that alloy D yields the highest hardness and Young's modulus. This enhanced hardness and Young's modulus of the alloy D may be attributed to stronger diffusional bond, highest density and structural integrity achieved in this alloy than that obtained in the other three alloys. Comparing the microstructural evidences in Fig. 3 with physical and mechanical property data presented in Figs. 6 and 7, it may be inferred that the maximum Young's modulus and hardness of the alloy D is obtained primarily due to the combined effect of dispersion hardening, grain refinement and solid solution hardening

(as Cr content in alloy D is the maximum). Solid solution hardening involves impending dislocation movement by dislocation-solute interaction. Similar effects were earlier reported by Timelli et al. [31] for AlSiCu(Fe) alloy, and Ma et al. [32] and Dybkov et al. [33] for Fe-3.5B alloys. Young's modulus and hardness values of the hydrostatically extruded products are summarized Table 1.

Fig. 8 shows the typical stress versus strain curves generated through compression tests of all the alloys with samples obtained along the longitudinal direction of the billets produced by hydrostatically extrusion at 1000 ^{o}C. The tests were repeated three times to ensure that the results were reproducible. Alloy D records the highest compressive strength (1660 MPa) with a true strain of 7.5%, while alloy A shows the lowest compressive strength (850 MPa) with a true strain of 22.5%. It is interesting to note that strength is directly proportional and ductility is inversely proportional to Cr content of the alloys. It may be recalled that SEM images in Fig. 3 showed that the grains in alloy A were elongated in longitudinal direction. This directionality was most pronounced in alloy A than in any of the other three alloys. It is known that that elongated grain structure enhances the ductility of extruded samples in longitudinal direction as compared to that for samples with polygonal recrystallized microstructure [34]. The lowest Cr containing alloy shows the highest ductility and the lowest strength. This trend is consistent with the results from earlier studies reported that the increase in Cr content of ferritic alloys increases the strength but reduces ductility [30]. In any case the combination of strength and ductility obtained in the present alloys is remarkable considering the fact that the alloys were produced through mechanical alloying followed by hot hydrostatic extrusion.

Fig. 9 shows the typical stress versus strain curves generated through compression tests of all the alloys using samples machined from the transverse direction of the hydrostatically extruded bars. The tests were repeated three times to ensure that the results were reproducible. The mechanical properties recorded in Fig. 9 are quite similar to that obtained from similar compression tests conducted with samples taken from longitudinal direction of all the extruded bars, except that the level of ductility is lower in the former tests. Alloy D shows highest compressive strength (2260 MPa) with a lower true strain of 5.0 % and alloy A records the lowest compressive strength (1240 MPa) with the highest true strain of 10.9 %. As already stated, the strength value is higher and elongation is lower in samples taken from transverse direction as compared to that taken from longitudinal extrusion direction in all the alloys. This is due to the difference in morphology or shape of the grains, i.e. smaller and circular geometry in transverse direction vis-à-vis larger and elongated in longitudinal direction, respectively. In the latter case, dislocations can glide up to a larger distance in grains elongated along direction of extrusion before encountering barriers from grain boundaries or dislocation tangles.

Relevant mechanical properties of all the four alloys are summarized in Table 1 for an overview and comparison as already discussed, these mechanical properties (hardness, Young's modulus, yield and compressive strength) increase with increase in Cr content in all the alloys and accordingly alloy D records the highest enhancement of mechanical strength albeit lowest ductility and alloy A shows precisely the opposite trend.

Enhancement of ductility depends on deformation mechanism, grain size and morphology, and orientation of the partially/fully recrystallized grain after hydrostatic extrusion. Inhomogeneous grain size distribution and morphology in extruded condition appear to be more conducive for enhancing ductility despite maintaining very high level of mechanical strength. The yield strength (σ_y) of polycrystalline materials increases from its base (σ_0) value (fully annealed and coarse grained state) as grain size decreases according to the well established Hall-Petch relationship [35]:

$$\sigma_y = \sigma_0 + Kd^{-1/2} \qquad (2)$$

where d is the average grain size and K is a constant.

The genesis of Hall-Petch strengthening is attributed to the barrier posed to dislocation movement by grain boundaries and enhancement of Peierl-Nabarro stress due to dislocation-dislocation interactions within the grain body. Many mechanisms and models were proposed and experiments conducted to rationalize this relationship; particularly with regard to nanostructured or ultrafine grain materials vis-à-vis their course grained counterparts. Indeed, extremely high strength and hardness have been reported in such nano-polycrystalline solids [36]. On the other hand, reversal of this trend is also noticed below a critical grain size reduction [37] for which a possible theoretical explanation for this inverse H-P relationship in nano-crystalline materials have been proposed [38, 39].

The elongation of polycrystalline materials depending on grain size is not well understood yet, because plastic instability is a complex process that can not be expressed by a simple relationship correlating only elongation and grain size [40]. However, in absence of an available comprehensive quantitative model on this subject, attempting an empirical or a semiempirical expression on physical model as proposed below:

The microstructure of the present alloys subjected to a uniaxial compressive load at room temperature may be schematically presented, as it is shown in Fig. 10. In this model, polycrystalline metallic matrix is strengthened mainly by grain refinement and by precipitation or dispersion of second phase particles/dispersoids located along boundaries or within the grains (Fig. 10a).

During plastic deformation the matrix is assumed to yield first, through dislocation movement and generating fresh lot of dislocations in the process [41] accumulated either around the second phase particles (i.e. dispersiods) or at the grain boundaries to accommodate the non-uniform deformation around these inhomogeneties (Fig. 10b). In the microscopic scale the plastic deformation is highly inhomogeneous. Due to the relative ease of yielding as enumerated by Hall-Petch relationship, the larger grains undergo earlier or larger extent of plastic deformation compared to smaller grains. The smallest grain might even behave some what like second phase particles offering strengthening rather than undergoing yielding themselves. Therefore, dislocations emitted from larger grains tend to accumulate around boundaries of smaller grains adjacent to the larger grains for accommodating the non-uniform deformation (Fig. 10b).

The following semi-empirical equation reported by Li et al. [40] may predict the influence of grain size (d) on the degree of elongation (ε) of grain by uniaxial deformation:

$$\varepsilon_u = \varepsilon_0 - \frac{1}{M + Nd} \ , \qquad (3)$$

where, ε_0, M and N are material parameters. This equation predicts that the uniform elongation increases with increasing grain size. It has been reported recently that ultrafine grained low carbon steels can be obtained by intense plastic straining [42-45]. Several

special intense staining processes, such as equal channel angular pressing, accumulative roll bonding, cold rolling, caliber warm rolling, and mechanical milling of powder metals have succeeded in producing ultafine grained steels [46-49]. Unlike these uniform deformation methods, the present approach of hydrostatic extrusion process produces non-uniform or perhaps bimodal grains so that the ultrafine defect-free matrix grains generated through dynamic recrystallization during hydrostatic extrusion itself can amount to strengthening equivalent to dispersion strengthening by large precipitates or dispersoids.

The effect of such dispersion hardening has already been evidenced by TEM bright field image (Fig. 5) where the size of smaller grains and mixed oxide dispersoids or precipitates particle are 20-40 nm and 10-20 nm, respectively. The corresponding combined strengthening mechanism from both dispersoids and ultra-fine grains fit well with Orowan-type mechanism [50], which explains the role of non-shearable particles in strengthening the matrix. The mechanism involves by-passing stress obtained by taking into account the influence of the dislocation character (edge or screw) on the equilibrium shape of the loop and interaction of the two arms of the dislocation on opposite sides of the loop. The release of dislocation is strongly depend on the obstacles shape and size in the Orowan loop. The detail mechanism of solid solution and dispersion or precipitation hardening in the present set of oxide dispersion strengthened by ferritic alloys was earlier discussed by Karak et al. [25, 26].

SEM images of fracture surfaces of all the alloys A to D are shown in Fig. 11(a-d). Fig. 11(a, b) reveal distinct evidences of ductile failure in alloys A and B marked by elongated dimples. In particular, the small and uniform dimples are revealed on the

fracture surface of hydrostatically extruded alloy A (Fig.11a) is remarkable evidence of ductility in a multi-component high alloy solid prepared through powder metallurgy processing. The higher the accumulated strain, greater is the overall grain boundary and sub-grain boundary area. Since grain boundaries were effective obstacles to dislocation motion, and fine grained alloy would have a higher density of grain boundaries per unit volume, the strength of alloy D increases with decreasing grain size. The remarkable improved ductility was obtained in the alloy A in both directions because elongated grains contributed to macroscopic deformation and the stress concentrations were accordingly reduced and spread over a wider area. Moreover, the hydrostatic extrusion process not only refined the grain size, but also changed the morphology of grains. The breakage or conversion of intermetallic particles into smaller fragments parts facilitated the dislocation motion and overall ductility of the alloy.

As suggested by Hawk et al. [51], higher plastic deformation and ductility can arise only through a dislocation climbing process over the intermetallic dispersoids in such dispersion strengthened matrix. Such interactions affect the matrix deformation behavior resulting in an increased flow stress but decreased capacity for strain hardening. The compressive strength of the current alloys is maximum for alloy D consolidated by hot isostaic pressing at 1000 ^{o}C as compared to the other two techniques, namely high pressure and pulse plasma sintering. The interesting thing is that the hot isostatic pressing [25, 26] and high pressure sintering [27] methods made the alloys brittle in nature which was proved in our earlier reports. But, the same alloys showed higher compressive strength with little ductility in pulse plasma sintering technique [24]. It is observed the

substantial improvement of ductility of the same set of alloys can now be achieved by hydrostatically extrusion technique. The comparison of mechanical properties in terms of hardness, Young's modulus, yield stress, compressive stress and fracture toughness of all the alloys sintered at 1000^{o}C for high pressure sintering, hot isostaic pressing, pulse plasma sintering and hydrostatic extrusion have been summarized in Table 2. It is clear that the maximum compressive strength, hardness, Young's modulus and fracture toughness have achieved in alloy D for all the processing routes. The beauty of the study is that the mechanical properties of same set of alloys are varying with respect to processing routes and its parameters. Thus, it is proved that mechanical property and deformation behavior depends on the processing techniques as well as processing parameters for solids developed by powder metallurgy route.

4. Conclusions

The present study suggests that hydrostatic extrusion is an extremely promising method way for consolidation of mechanically alloyed powders of nano-Y_2O_3 dispersed Fe-Cr-Al-Ti ferritic alloys. Density, hardness and compressive strength of all the alloys increase with increase in Cr content. The present ferritic alloys record significantly high compressive (1240-2226 MPa) and yield strength (1025-1505 MPa) with a response high true strain (10.9-5.0 %) in transverse direction and reasonably high compressive (850-1660 MPa) and yield strength (525-1094 MPa) with a significantly high level of true strain (22.5-7.5 %) in longitudinal direction. In addition, the Young's modulus (240-265 GPa) and hardness (14.7-17.8 GPa) values are also very high and measure up to 1.5 times with a lower density (~ 7.4 Mg/m^3) than that of other oxide dispersion strengthened

ferritic alloys (< 1200 MPa). The novelty of the present consolidation route lies in the unique microstructure comprising uniform distribution of 20-30 nm $Y_2Ti_2O_7$ or Y_2O_3 particles, essential for grain boundary pinning and creep resistance, and dispersed in large matrix grains (ferritic) with substantial solid solution strengthening. Despite, the extremely high strength, significant level of ductility recorded in the present alloys as compared to that obtained from the same set of alloys consolidated by high pressure sintering [24], hot isostatic pressing [25, 26], pulse plasma sintering [29] earlier reported by us, can be attributed to more effective sintering of the powder mass and greater structural integrity, isotropy, density and ductility achieved by the present consolidation method (hydrostatic extrusion) than earlier techniques.

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Figure Captions

Fig. 1: (a) Schematic representation of the apparatus used for hydrostatic extrusion [18, 19] and (b) sample after hydrostatic extrusion

Fig. 2: XRD patterns after different stages of mechanical alloying and hydrostatic extrusion at 1000°C after 40 h of mechanical alloying for (a) alloy A, (b) alloy B (c) alloy C and (d) alloy D, respectively

Fig. 3: SEM images of the hydrostatically extruded bar of (a) alloy A, (b) alloy B, (c) alloy C and (d) alloy D (transverse section) respectively. For comparison similar

SEM images of (e) alloy A and (f) alloy D taken from (longitudinal direction) are also presented.

Fig. 4: Results on grain size distribution from image analysis of the SEM images from the cross sectional (transverse) view of (a) alloy A, (b) alloy B, (c) alloy C and (d) alloy D, respectively

Fig. 5: Bright field TEM image of alloy A hydrostatically extruded at 1000^{o}C: (a) low (b) high magnification view of transverse section, and (c) the same along longitudinal direction

Fig. 6: Variation of density (solid symbols) and porosity (open symbols) as a function of Cr content (wt %) after hydrostatic extrusion at 1000°C

Fig. 7: Variation of hardness (solid symbols) and Young's modulus (open symbols) as a function of Cr content (wt %) after hydrostatic extrusion at 1000° C

Fig. 8: Variation of true stress versus true strain during uniaxial compression testing of samples from all four alloys obtained along the longitudinal direction of the billet after hydrostatic extrusion at 1000^{o}C

Fig. 9: Variation of true stress versus true strain during uniaxial compression test of samples from all four alloys obtained along the transverse direction of the billet after hydrostatic extrusion at 1000^{o}C

Fig. 10: Schematic of (a) larger and smaller grains along with second phase particles/dispersiods and (b) Dislocations generation at grain boundaries and around second phase particles/dispersiods

Fig. 11: FESEM fractographic images after failure of hydrostatically extruded samples subjected to uniaxial compression of (a) alloy A (b) alloy B, (c) alloy C and (d) alloy D, respectively.

Table Captions

Table 1: Summary of mechanical properties of alloys A, B, C and D hydrostatically extruded (at 1000 °C) condition

Table 2: Comparison the mechanical properties of the alloys sintered at 1000 °C for high pressure sintering, hot isostatic pressing, pulse plasma sintering and hydrostatic extrusion