Evaluation of Microstructure and Mechanical Properties of Nano-Y$_2$O$_3$ Dispersed Ferritic Alloy Synthesized by Mechanical Alloying and Consolidated by High Pressure Sintering

S. K. Karak[1], J.Dutta Majumdar[2], Z. Witczak[3], W. Lojkowski[3], L. Ciupinski[4], K. J. Kurzydłowski[4] and I. Manna[2,5] *

[1]Metallurgical and Materials Engineering Department, National Institute of Technology, Rourkela 769008, India

[2]Metallurgical and Materials Engineering Department, Indian Institute of Technology, Kharagpur 721302, India

[3]Institute of High Pressure Physics (Unipress), Polish Academy of Sciences, Sokolowska 29, 01-142 Warsaw, Poland

[4]Faculty of Materials Science and Engineering, Warsaw University of Technology, Wołoska 141, 02-507 Warsaw, Poland

[5]CSIR-Central Glass and Ceramic Research Institute, 196 Raja S C Mullick Road, Kolkata 700032, India

Abstract

In this study, an attempt has been made to synthesize 1.0 wt % nano-Y$_2$O$_3$ dispersed ferritic alloys with nominal compositions: 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) steels (all in wt %) by solid state mechanical alloying route and consolidation the milled powder by high pressure sintering at 873 K (600°C), 1073 K (800°C) and 1273 K (1000°C) using 8 GPa uniaxial pressure for 3 min. Subsequently, an extensive effort has been undertaken to characterize the microstructural and phase evolution by X-ray diffraction, scanning and transmission electron microscopy and energy dispersive spectroscopy. Mechanical properties including hardness, compressive strength, Young's modulus and fracture toughness were determined using micro/nano-indentation unit and universal testing machine. The present ferritic alloys record extraordinary levels of compressive strength (1150-2550 MPa), Young's modulus (200-240GPa), indentation fracture toughness (3.6 to 15.4 MPa√m) and hardness (13.5-18.5 GPa) and measure up to 1.5-2 times greater strength but with a lower density (~ 7.4 Mg/m^3) than other oxide dispersion strengthen ferritic steels (< 1200 MPa) or tungsten based alloys (< 2200 MPa). Besides superior mechanical strength, the novelty of these alloys lies in the unique microstructure comprising uniform distribution of either nanomertic (~10 nm) oxide (Y$_2$Ti$_2$O$_7$ / Y$_2$TiO$_5$ or un-reacted Y$_2$O$_3$) or intermetallic (Fe$_{11}$TiY and Al$_{9.22}$Cr$_{2.78}$Y) particles ferritic matrix useful for grain boundary pinning and creep resistance.

*Author for correspondence Email: imanna@metal.iitkgp.ernet.in; Fax: +91 3222 255303, Tel: +91 3222 283266

I. INTRODUCTION

Ferritic alloys are considered a suitable candidate for heat resistant structural applications in nuclear and thermal power plants and fast breeder reactors due to the unique combination of body center cubic structure with good swelling resistance, low co-efficient of thermal expansion, high thermal conductivity, good oxidation and creep resistance and high tensile/compressive strength. However, utility of ferritic alloy is limited to temperatures of up to 823 K (550°C) due to lack of or inadequate creep strength above that temperature. A possible strategy to improve strength at room temperature and elevated temperatures is to strengthen the matrix by uniform dispersion of nanometric oxide that would prevent grain boundary sliding. Oxide dispersion strengthened steels produced by mechanical alloying with Y_2O_3 dispersion have recorded higher creep strength compared to traditional ferritic/martensitic steels/alloys of similar compositions [1–4]. Oxide dispersion strengthening in metallic alloys is more effective when the dispersoids are small and uniformly dispersed in the ferrite matrix. It has been demonstrated that oxide-dispersion strengthened ferritic steels/alloys can maintain good mechanical properties at elevated temperatures and have greater swelling resistance than the austenitic steels [5–14]. Mechanical alloying is a solid state processing route to synthesize alloys from elements/components possessing widely different physical/chemical/thermodynamic properties that could pose evaporation/segregation/inhomogeneity related problems while processing through melting/casting route. Mechanical alloying enables uniform dispersion of nano-sized oxide particles such as yttria (Y_2O_3) in the ferritic matrix. However, the product is alloyed powder (not a bulk component) with novel microstructure including extended solid solution in nanostructured or amorphous state. In order to assess the mechanical properties and study the deformation mechanism, these powders need to be consolidated into solid components with adequate density and integrity by a suitable technique that allows retention of the as-milled microstructure and phase aggregate. In the past, several techniques have been explored to consolidate the mechanically alloyed powder e.g., hot extrusion [15–17], equichannel angular extrusion [18-20],

hot pressing [21, 22], cold consolidation using severe plastic deformation [23, 24] and hydrostatic extrusion [25]. Recently, sintered products with high density have been obtained by high-pressure sintering, which involves the application of very high uniaxial pressure (up to 8 GPa) at an elevated temperature only for a short period (~1–3 min) of time [26, 27]. The primary advantage of this sintering route lies in the application of very high pressure only for limited time at a very high temperature for densification of the powders. Recently, we have reported consolidation of the same set of mechanically alloyed ferritic alloys by hot isostatic pressing at 873-1273 K (600-1000 °C) under 1.2 GPa uniaxial pressure [28, 29] and pulse plasma sintering at 873-1273 K (600-1000 °C) using 75 MPa uniaxial pressure applied for 5 min and 70 kA pulse current at 3 Hz pulse frequency [30].

The present investigation aims to synthesize nano-Y_2O_3 dispersed Fe-Cr-Al-Ti ferritic alloys by mechanically alloying/milling, consolidate the milled powder by high pressure sintering at 873-1273 K (600-1000 °C) and assess physical (density and porosity) and mechanical properties (hardness, Young's modulus, compressive strength, indentation fracture toughness) of the alloys to establish the structure-property-process parameter correlation.

II. EXPERIMENTAL PROCEDURE

Appropriate amounts of pure Fe, Cr, Al, Ti and Y_2O_3 powder, each constituent having ≥ 99.5 wt % purity with particle size of about 50-100 μm, were subjected to mechanical alloying in high-energy planetary ball mill with 10:1 ball to powder mass ratio in stainless steel container with 10 mm diameter stainless steel balls to yield four different single phase Fe-Cr-Al-Ti alloys with 1.0 wt % nano-Y_2O_3 dispersion at room temperature. The chemical composition of powder blends subjected to mechanical alloying is summarized at Table 1. Milling was carried out in wet (toluene) medium to prevent agglomeration of the powders and to retard oxidation beyond the

initial stage of milling. The identity and sequence of the phase evolution at different stages of mechanical alloying were studied by X-ray diffraction (XRD) using Co-K_α (0.707 nm) radiation and scanning (SEM) and transmission electron microscopy (TEM). The alloy powder was pre-compacted under 50 MPa pressure in vacuum (4.5×10^{-4} mbar) using manual press. The cold pressed cylindrical preforms of 7 mm diameter and 4-5 mm height were placed in special $CaCO_3$ ceramic containers, positioned between tungsten carbide anvils with graphite susceptor coil surrounding the crucible and sintered at 873 K (600°C), 1073 K (800°C) and 1273 K (1000°C) under 8 GPa uniaxial pressure. While the pressure was gradually increased to 8 GPa, heating through the graphite susceptor was applied only for 3 min. This transient heating at extremely high pressure produced high density product. Following sintering, the samples (7.0 mm diameter cylindrical compacts) were allowed to cool to room temperature inside the ceramic die and subjected to subsequent characterization.

Morphology, size, shape and distribution of the phases in the powders or sintered components and surface damage in worn tracks were studied using a field emission gun assisted scanning electron microscopy (FESEM, make: Carl Zeiss, Supra 40 V). Electron transparent thin foils were prepared for transmission electron microscopy studies initially by mechanical polishing followed by argon ion thinning using a GATAN precision ion mill for about 30 min. Selected foils prepared from alloys sintered at 1273 K (1000 ^{o}C) were examined under a high resolution transmission electron microscope (HRTEM) operated at 200 kV, JEM 1200 (JEOL, 200 kV) with double spherical aberration correctors and SEM Hitachi S-5500, using both bright and dark field as well as high resolution lattice imaging mode. Selected area diffraction (SAD) analysis was conducted to identify the phases present in the milled/sintered product. Qualitative information on chemical compositions at different locations was obtained using the energy dispersive spectroscopy (EDS) units (Oxford, UK) equipped with an ultra-thin window and attached to both the FESEM and HRTEM.

The density and porosity of milled powders and sintered products were determined by using a helium pycnometer (AccuPyc 1330) [31]. The average hardness of the samples was measured by using standard nano-indentation hardness (TriboIndenter with MultiRange NanoProbe, Hysitron) tester at 200 mN load. Each hardness value reflects an average of 25 individual measurements by nano-indentation at equivalent locations. The sintered samples were indented using Vickers micro-hardness tester (Leica, USA) with loads in the range of 1-20 kgf applied for a dwell time of 15 s. The lengths of cracks at the corner of indentations were measured at room temperature by analyzing the FESEM images to estimate the indentation fracture toughness. As the crack lengths exceeded three times the length of half-diagonal of their corresponding indentation, the empirical relationship proposed by Nihara et al. [32] for median cracks was employed to determine the fracture toughness (K_{1C}) as follows:

$$K_{1C} = 0.010(\frac{E}{H})^{\frac{2}{3}}(\frac{P}{C^{\frac{3}{2}}})$$

(1)

Where, E is the Young's modulus, H is the micro-hardness, P is the load used for indentation and C is the characteristic crack length, respectively. Similar indentation studies with the same set of alloys in hot isostatic pressing were recently reported by Karak et al. [29].

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 in a 10 kN universal testing machine with tungsten carbide anvils operated at 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 a LVDT an extensometer with an accuracy of ± 1.0 µm. During the compression test, the commencement of micro-cracking was monitored by acoustic emission method [33]. The fracture surfaces after compression tests were studied using FESEM with EDS.

III. RESULTS AND DISCUSSION

Figures 1 to 4 show the XRD patterns of the four different powder blends, listed in Table 1 as alloys A, B, C, and D, respectively. These blends were subjected to mechanical alloying for different cumulative time periods (0 to 40 h).

It is apparent that the milled product 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 30-40 h. The added Y_2O_3 (1.0 wt %) was too small to produce separate peaks. Furthermore, the increase in full width at half maximum ($\Delta\theta$) with milling time suggests that both crystallite size reduction and plastic strain accumulation are significant. Careful analysis of $\Delta\theta$ of the most prominent peak in a given XRD profile allows determination of plastic strain and crystallite size using the standard peak broadening analysis procedure based on Scherrer equation after elimination of contributions from the strain and instrumental error [34]. This exercise, presented in Figure 5, shows the variation of accumulated plastic strain and crystallite size as a function of milling time for all the four alloys. It is apparent that the crystallite size reaches truly nanometric level within 10 h of mechanical alloying. It may be noted that equilibrium solubility limit of Cr and Ti in α-Fe is 9.19 wt% and 0.0005 wt% (approximately), respectively; Al may dissolve in α-Fe up to 32.6 wt%. Perhaps, substantial reduction of crystallite size of the solvent (α-Fe) aids easy dissolution of all solute atoms (Cr, Ti and Al) and formation of single phase solid solution due to Gibbs-Thompson effect [35]. It is apparent that decrease in crystallite size and increase in residual strain of all the alloys is influenced by Cr content. The BET surface area and true powder density of the alloys A, B, C and D are summarized in Table 2. It may be noted that the surface area gradually increases with increasing Cr content of the alloys. Figure 6 (a, b) and Figure 7 (a, b) show the typical bright field TEM image and corresponding selected area diffraction (SAD) pattern of the 40 h milled powders of alloy A and D, respectively. The powder particles contain nanocrystalline BCC-Fe grains with intermetallic phase and mixed oxide phase dispersed in the matrix. The corresponding SAD pattern shows diffraction rings that

can be indexed as the (110), (200) and (211) planes of the BCC Fe-Cr phase, (222) plane of the $Y_2Ti_2O_7$ or (201) plane of the Y_2TiO_5 mixed oxide phase and (222) of the un-reacted Y_2O_3 phase, respectively. The calculated interplanar spacings (d) of BCC Fe-Cr phase matches with the standard d-values of 0.2020 nm for (110), 0.1430 nm for (200) and 0.1010 nm for (220) planes, respectively. Similarly, the measured d-values match with the standard values of 0.2914 nm for (222) of $Y_2Ti_2O_7$ and 0.3012 nm for (201) of Y_2TiO_5 mixed oxide, respectively. The weak-intensity inner ring ($d = 0.3062$ nm) in Figure 6b can be attributed to (222) of un-reacted (ex-situ) Y_2O_3 phase.

The interplanar spacing (d) calculated form one set of rings in both Figure 6b and Figure 7b match with those of $Y_2Ti_2O_7$ [36]. Thus, Figure 6 and Figure 7 confirm the presence of 10-20 nm $Y_2Ti_2O_7$ / Y_2TiO_5 or unreacted Y_2O_3 particles distributed uniformly in the BCC-Fe(Cr) matrix synthesized by the present mechanical alloying routine. It may be mentioned that presence/detection of $Y_2Ti_2O_7$ instead of Y_2O_3 in the SAD patterns suggests that Ti-ion has possibly penetrated into and partially substituted Y in Y_2O_3 to form the $Y_2Ti_2O_7$ / Y_2TiO_5 mixed oxides, besides retaining some un-reacted Y_2O_3 pure oxide during mechanical alloying.

Figure 8 to Figure 11 show the XRD pattern of the alloys A, B, C and D following high pressure sintering at 873 K (600°C), 1073 K (800°C) and 1273 K (1000°C), respectively. It appears that BCC-Fe(Cr) phase is the predominant constituent of the sintered product along with intermetallic phases like $Fe_{11}TiY$ and $Al_{9.22}Cr_{2.78}Y$, in addition to the presence of mixed oxide phase $Y_2Ti_2O_7$ / Y_2TiO_5 or un-reacted Y_2O_3. It may be noted that the presence of $Y_2Ti_2O_7$ / Y_2TiO_5 or unreacted Y_2O_3 was already noted in the mechanically alloyed product prior to sintering (Figures 6, 7). The other two phases have formed during sintering at 873-1273 K (600-1000 °C) under high pressure.

Figure 12 shows he bulk EDS analysis of alloy A sintered at 1273 K (1000°C), confirms the presence of elements Fe, Cr, Al, Ti, Y. Table 3 presents the summary of EDS analysis of all the four alloys in mechanically alloyed (40 h) and sintered conditions. Besides confirming the

presence of elements Fe, Cr, Al, Ti and Y, the results suggest that the final milled/sintered products retain practically the same composition as the initial powder blend without incurring any major materials loss due to oxidation or dilution due to impurity pick up.

Figures 13 (a-c) shows the bright field and dark field TEM image and the corresponding SAD of alloy A sintered at 873 K (600°C). Figure 13a evidences the presence of 10-20 nm ultrafine particles distributed uniformly in the matrix. Figure 13c confirms these nanomatric particles as $Y_2Ti_2O_7$ / Y_2TiO_5 or un-reacted Y_2O_3 embedded in the BCC-Fe(Cr) matrix. Figure 14 (a, b) show the bright field and corresponding SAD pattern of alloy A sintered at 1273 K (1000°C). It is apparent that increase in sintering temperature from 873 K (600 $^{\circ}$C) to 1273 K (1000°C) leads to substantial grain growth.

Figure 15 shows the variation of density and porosity as a function of sintering temperature for the four different alloys. As sintering temperature increases the density increases and porosity decreases in all the present alloys. It is apparent that the maximum density is attained by sintering at the highest temperature (1273 K (1000°C)) in all the four alloys. By the same logic, the lowest density is obtained by consolidation at the lowest temperature (873 K (600°C)). This is due to incomplete grain bridge formations or welding at low temperature. Similar kind of results were obtained from the same set of ferritic alloys by hot isostatic pressing at 873-1273 K (600-1000 °C) under 1.2 GPa uniaxial pressure by Karak et al. [29] and pulse plasma sintering at 873-1273 K (600-1000 °C) using 75 MPa uniaxial pressure applied for 5 min and 70 kA pulse current at 3 Hz pulse frequency by Karak et al. [30]. Beside such results from our group [29, 30], similar results on the role of temperature in sintering of mechanically alloyed powders in other alloy system were also reported, e.g. by Roy et al. [37] in $Al_{65}Cu_{20}Ti_{15}$ alloy, Mao-lin et al. [38] in nano-SiC, Yang et al. [39] in WC-ZrO_2-VC ceramic composites, Zhaohui et al. [40] in cubic boron nitride and Ardestani et al. [41] in W-20-40 wt% Cu composite powder.

Figure 16 shows the variation of the Young's modulus and hardness of the mechanically alloyed and sintered products of all the four alloys as a function of the sintering temperature. It is

apparent that high pressure sintering of all the alloys at 1273 K (1000 ^{o}C) yields the highest hardness and Young's modulus. This enhanced strength at higher temperature may be attributed to stronger diffusional bond and structural integrity achieved due to greater diffusional activity, despite grain growth, at 1273 K (1000 ^{o}C) than that obtained at 873 K (600 ^{o}C) or 1073 K (800 ^{o}C). Based on the observations from the results presented in Figures 15 and 16, it is possible to infer that the maximum Young's modulus and hardness is obtained under the processing condition that yields the highest density. Obviously, the density, Young's modulus and hardness of the alloys sintered at low temperature (say, 873 K or 600 ^{o}C) are lower for the condition that yields incomplete densification. However, the increase in hardness and Young's modulus on pressing at the higher temperature can probably be attributed to the densification of matrix as well as pinning of the grain boundary by the uniform distribution of $Y_2Ti_2O_7$ / Y_2TiO_5 or un-reacted Y_2O_3. Very similar trend of results was obtained of the same set of ferritic alloys by hot isostatic pressing at 873-1273 K (600-1000 °C) under 1.2 GPa uniaxial pressure by Karak et al. [29] and pulse plasma sintering at 873-1273 K (600-1000 °C) using 75 MPa uniaxial pressure applied for 5 min and 70 kA pulse current at 3 Hz pulse frequency by Karak et al. [30]. Identical trend was reported in other alloy systems as quoted earlier with regards to density and porosity, by Roy et al. [37] in $Al_{65}Cu_{20}Ti_{15}$ alloy, Mao-lin et al. [38] in nano-SiC, Yang et al. [39] in WC-ZrO_2-VC ceramic composites, Zhaohui et al. [40] in cubic boron nitride and Ardestani et al. [41]] in W-20-40 wt% Cu composite powder.

Figure 17 shows the typical stress versus displacement curves generated through compression tests of the alloy A consolidated by high pressure sintering at different temperatures. The tests at each temperature were repeated three times to ensure that the results were reproducible. From the stress-displacement curves, it is obvious that failure has occurred in the elastic regime itself prior to yielding indicating that ductility under compression is almost non-existent. The maximum compressive strength of 2550 MPa was obtained in the alloy D sintered at 1273 K (1000 ^{o}C). These results of mechanical properties are summarized at Table 4.

The process of fracture initiation and propagation has been independently monitored through the observation of the cumulative intensity of acoustic emission events recorded from the unit volume of the samples subjected to compressive loading (Figure 18). It may be noted that the stress corresponding to the initiation of failure is distinguished by the rapid increase of acoustic emission activity marked by arrowheads (Figure 18). It is worth noting that the extent of elastic deformation prior to failure and the load bearing capacity of the sample pressed at 1273 K (1000 ^{o}C) is higher than in those sintered at lower temperatures. It may also be noted that the compressive strength and hardness of these current alloys are 1.5 to 2 times higher than that ($\approx$ 1400 MPa) of oxide dispersion strengthened MA 957 (Fe-14Cr-1.0 Ti -0.3Mo- 0.25 Y_2O_3) steel those reported by Kluch et al. [42] and also higher than that ($\approx$ 1200 MPa) of nano-Y_2O_3 dispersion strengthened Ni-based superalloy [43]. The compressive strength of the present alloys with a low density have been found greater than that of the maximum compressive strength ($\approx$ 2203 MPa) of the tungsten alloy (93W-4.9Ni-2.1Fe (wt %)) reported by Zhang et al. [44]. Lee et al. [45] have achieved maximum compressive strength of partially stabilized zirconia (PSZ) dispersed ODS tungsten heavy alloys (93W-5.6 Ni-1.4Fe (wt %)) to the tune of 1000 MPa which is much lower than that of the present ferritic alloys.

The significantly high compressive strength of the present alloys can be attributed to solid solution strengthening, precipitation strengthening and dispersion hardening. In the former mechanism, the foreign/solute atoms produce lattice strain that can impede the dislocation movement. Both elastic modulus and long range interactions are important components in this mechanism at low temperature. It may be noted that the hardness and compressive strength of all the four present alloys gradually increases with increasing content of Cr. The results from similar studies by Cacres et al. on Mg-Al alloy [46] and Mg-Zn alloy [47] corroborate this theory. On the other hand, ultrafine second phase particles (nano-$Y_2Ti_2O_7$ / Y_2TiO_5 or unreacted Y_2O_3) produced after consolidation have a low or no solubility in the matrix and can induce strength in the matrix by dispersion hardening mechanism. The strengthening mechanism of the same set of ferritic

alloys was reported recently in Karak et al. [29, 30] and similar kind of ODS alloys in Schneibel et al. [48, 49]. It was observed that the alloys were brittle in nature after hot isostatic pressing with 1.2 GPa uniaxial pressure at 873-1273 K (600-1000 °C) [29] and registered slightly improve ductility after pulse plasma sintering at 873-1273 K (600-1000 °C) with 75 MPa uniaxial pressure applied [30].

Figure 19 shows the FESEM image of alloy D sintered at 1273 K (1000 $^{\circ}$C) after indentation with 20 kgf load. The fracture toughness calculation is done from equation (1) by measuring the average crack length. The length of the crack is the minimum in all the alloys sintered at higher temperature (1273 K or 1000 $^{\circ}$C). It is already demonstrated that the density, hardness and compressive strength of the present alloys are a function of sintering temperature. Similar trend is noted with regard to average indentation fracture toughness of all the four alloys, as summarized in Table 4. The much lower indentation fracture toughness of the alloys processed at 873 K (600 $^{\circ}$C) is believed to be due to its higher porosity content. An FESEM image depicting a typical indentation crack recorded under a load of 20 kgf is shown in Figure 19.

The fracture surfaces formed due to compression tests are shown in Figure 20 at different magnifications. These FESEM images of the fracture surfaces reveal evidences of primarily brittle failure, which is obvious from the corresponding stress - displacement plots in Figure 17 and acoustic emission record of the compression test distinctly marking (Figure 18) the onset of failure at specific stress levels

Figure 20a shows evidence of cleavage type of failure as well as secondary cracking. On the other hand, examination of the fractograph in Figure 20b shows presence of nano-size particles on the fracture surface, suggesting that failure involves interfacial decohesion of the $Y_2Ti_2O_7$ / Y_2TiO_5 or unreacted Y_2O_3 particles from the Fe-Cr alloy matrix. Thus, either lack of adequate plastic activity in the matrix phase or decohesion at the dispersion-matrix interface could account for early failure of these alloys despite very high yield stress under compression. For ensuring reliability as a structural component, more effective sintering and ductility or

toughness should be the next target of development of these otherwise high strength alloys. In terms of fractography of the failure components , the present alloys showed quite similar behavior as that earlier reported from hot isostatic pressing at 873-1273 K (600-1000 °C) under 1.2 GPa uniaxial pressure [29].

The compressive strength of the current alloys is minimum for alloy D (Table 5) consolidated by high pressure sintering at 1273 K (1000 ^{o}C) as compared to that obtained after consolidation by other two techniques namely, hot isostaic pressing and pulse plasma sintering. It is important to note that for same set of alloys showed the mechanical properties in terms of hardeness, Young's modulus, compressive strength and indentation fracture toughness slightly lower side as compared to our earlier studies showed that the same alloys following sintering by hot isostatic pressing [28, 29] and pulse plasma sintering [30] were very strong but fairly brittle. However, the same alloys seem to yield comparable or higher compressive strength with marginally higher ductility following consolidation by pulse plasma sintering. Thus, it is interesting to note that the same set of mechanically alloyed powders with identical composition and microstructural state can eventually produce widely different mechanical properties when sintered by different techniques using the optimum process parameters.

IV. SUMMARY AND CONCLUSIONS

From, the detailed structural characterization and mechanical property assessment of the present nano- Y_2O_3 dispersed ferritic alloys the following conclusions are drawn:

1. The present ferritic alloys record extraordinary high levels of compressive strength (1150-2550 MPa), Young's modulus (200-240GPa), indentation fracture toughness (3.6 to 15.4 MPa√m) and hardness (13.5-17.5 GPa) and measure up to 1.5-2 times greater strength with a lower density (~ 7.4 Mg/m^3) than other oxide dispersion strengthen ferritic steel (< 1200 MPa) or tungsten based alloys (< 2200 MPa).

2. The novelty of these alloys lie both in the compositional range (wider range of Fe and Cr contents) and microstructural features (uniform nanometric (10-20 nm) dispersion of ex-situ pure (un-reacted Y_2O_3) and in-situ ($Y_2Ti_2O_7$ / Y_2TiO_5) mixed oxides and/or in-situ intermetallic phases ($Fe_{11}TiY$ or $Al_{9.22}Cr_{2.78}Y$). The latter is useful for dispersion strengthening and/or grain boundary pinning at elevated temperature.

3. Despite high mechanical strength, ductility or toughness of the present alloys sintered by high pressure sintering is not satisfactory as yet. The present results are similar to that reported earlier from the same set of alloys sintered by hot isostatic pressing and pulse plasma sintering.

REFERENCES

[1]   R. K. Nanstad, D. A. McClintock, D. T. Hoelzer, L. Tan, T. R. Allen J. Nucl. Mater., 2009, vol. 392, pp.331-40.

[2]   S. Ohtuska, S. Ukai, M. Fujiwara, T. Kaito, T. Narita, Mater. Trans. A, 2005, vol. 46, pp. 487-92.

[3]   S. Ukai, S. Mizuta, T. Yoshitake, T. Okuda, M. Fujiwara, S.Hagi, T. Kobayashi, J. Nucl. Mater., 2000, vol. 283–87, pp.702-06.

[4]   M. K. Miller, D. T. Hoelzer, E. A. Kenik, K. F. Russell, Intermetallics, 2005, vol.13, pp. 387-92.

[5]   R. W. Evans, J. Presto, B. Wilshire, E. A. Little, J. Nucl. Mater. 1992, vol.195, pp. 24.

[6]   S. Ukai, M. Harada, H. Okada, M. Inoue, S. Nomura, S.Shikakura, K. Asabe, T.   Nishida, M. Fujiwara, J. Nucl.Mater. 1993, vol. 204, pp. 65-73.

[7]   C. Zankine, C. Prioul, D. Francois, Mater. Sci. Eng. A. 1996, vol. 219, p. 102.

[8]   D. K. Mukhopadhyay, F.H. Froes, D.S. Gelles, J. Nucl.Mater. 1998, vol. 258–63, pp. 1209-15.

[9]   S. Ukai, T. Nishida, T. Okuda, T. Yoshitake, J. Nucl. Mater. 1998, vol. 258–63, pp. 1745-49.

[10]   I. S. Kim, J. D. Hunn, N. Hashimoto, D. L. Larson, P. J.Maziasz, K. Miyahara, E. H. Lee, J. Nucl. Mater. 2000, vol. 280, pp. 264-74.

[11]   C. Capdevila, Y. L. Chen, N. C. Krieger Lassen, A. R. Jones and H.K. D. H. Bhadeshia: ISIJ International,2003,vol. 43, pp. 777–83.

[12]   C. P. Jongenburger and R. F. Singer. In: E. Arzt and L. Schultz, Editors, Proc. Conf. New Materials by Mechanical Alloying Techniques, Calw-Hirsau, DGM Verlag Oberursel (1988) p. 157

[13]   K. Murakami, H. Harada and H. K. D. H Bhadeshia, Grain Boundary Topology in Mechanically Alloyed MA6000. Proceedings of Heat Treatment '92", edited by I. Tamura, Kyoto, Japan, (1992), pp. 269-72.

[14]   T. S. Chou, H. K. D. H Bhadeshia, G. McColvin and I. C. Elliott, Atomic Structure of mechanically alloyed steels. In: Mechanical Alloying for Structural Applications. Proceedings of the 2nd International Conference on Structural Applications of Mechanical Alloying. Vancouver, British Columbia, Canada, Ed: ASM (Materials Park, Ohio, USA) (1993) pp. 77-82.

[15]   J. Q. Guo, K. Kita, N. S. Kazama, J. Nagahora, K. Ohtera, Mater. Sci. Eng. A, 1995, vol. 203, pp. 420–26.

[16]   K. I. Moon, K. S. Lee, J. Alloy Compd., 1999, vol.291, pp.312–21.

[17]   S. J. Hong, B. S. Chum, Mater. Sci. Eng. A., 2003, vol. 348, pp. 262–70.

[18]   O. N. Senkov, S. V. Senkova, J. M. Scott, D.B. Miracle, Mater. Sci. Eng. A, 2005, vol. 393, pp.12–21.

[19]   S. Z. Han, M. Goto, C. Lim, C. J. Kim, S. Kim, J. Alloy Compd., 2007, vol. 434-35, pp. 304-6.

[20]   S. Bera, Z. Zuberova, R .J. Helling, Y. Estrin, I. Manna, Philos. Mag., 2010, vol. 90, pp. 1465-83.

[21]   Y. Kawamura, H. Mano, A. Inoue, Scripta Mater., 2001, vol.44, pp. 1599–1604.

[22]   I. Borner, J. Eckert, Scripta Mater., 2001, vol. 45, pp. 237–44.

[23]   A. R. Yavari, W.J. Botta-Filho, C.A.D. Rodrigues, C. Cardoso, R.Z. Valiev, Scripta Mater., 2002, vol.46, pp. 711–16.

[24]   V. M. Segal, Mater. Sci. Eng. A, 2002, vol. 338, pp. 331–44.

[25]   W. Pachla, M.Kulczyk, M. Sus-Ryszkowska, A. Mazur and K. J. Kurzydlowski, J. Mater. Proce. Tech., 2008, vol. 205, pp. 173-82.

[26]   S. Bera, W. Lojkowsky, I. Manna, Metall. Mater. Trans. A, 2009, vol. 40, pp. 3276-83

[27]   D. Roy, R.Mitra, T.Chudoba, Z.Witczak, W.Lojkowski I. Manna, Mater. Sci. Eng. A, 2008, vol. 497, pp. 93–100

[28]   S. K. Karak, C.S.Vishnu, Z. Witczak, W. Lojkowski, J. Dutta Majumdar and I. Manna, Wear, 2010, vol.270, pp.5-11

[29]   S. K. Karak, T. Chudoba, Z. Witczak, W. Lojkowski and I. Manna, Mater. Sci.Engg A., 2011, vol.528,pp.7475-83

[30]   S. K. Karak, J. Dutta Majumdar, W. Lojkowski, A. Michalski, L. Ciupinski, K. J. Kurzydlowski and I.Manna, (2011) Philos.Mag., 2012, vol.92, pp.516-34

[31]   A. Presz, M. Sikibska, M. Pilecki, Powd. Hand. & Proc., 1995, vol. 7, pp. 321-25

[32]   K. Nihara, R. Morena, D.P.H Hasselman, Indentation fracture toughness of brittle materials for palmqvist cracks. In: Bradt RC, Hasselman DPH, Lange FF, editors. Brittle matrix composites 2. New York: Elsevier Applied Science (1983) pp. 84-97

[33]   Z. Witczak, Mater Sci Eng. A., 1997, vol. 239, pp. 206-12

[34]   T. H. Keijser, I. L. Langford, E. J. Mittemeijer, B. P. Vogel, J. Appli. Crystallogr., 1982, vol. 15, pp. 308-14.

[35]   David A. Porter and Kenneth E. Easterling, Phase Transformation in Metals and Alloys, Van Nostrand Reinhold Co. Ltd, New York, NY 10020, USA,(1981) pp. 44-47

[36]   L. F. He, J. Shirahata, T. Nakayma, T. Suzuki, H. Suematsu, I. Ihara, Y. W. Bao, T. Komastu and K. Niihara, Scripta Materialia, 2011, vol.64, pp.548-51

[37]   D. Roy, D. Chakravarty, R. Mitra, I. Manna, J. Alloy Compd., 2008, vol. 460, pp. 320–25.

[38]   Xie Mao-lin, Luo Du-li, Xian Xiao-Bin, Leng Bang-yi, Chen Chang'an , Lu Wei-yuan, Fusion Eng & Des., 2010,vol.85, pp. 964-68

[39]   F. Z. Yang, J. Zhao, X. Ai, J. Mater. Proc. Tech., 2009, vol.209, pp. 4531–36.

[40]   Y. Zhao, M. Wang, J. Mater. Proc. Tech., 2009, vol. 209, pp. 355–59

[41]   M. Ardestani, H.R. Rezaie, H. Arabi, H. Razavizadeh, Int. J. Refractory Metals and Hard Materials., 2009, vol. 27, pp. 862–67.

[42]   R.L. Klueh, J. P Shingledecker, R.W.Swindeman, D. T. Hoelzer, J. Nucl. Mater., 2005, vol. 341, pp.103–14

[43]   X. D. He, Y. Xin, M. W. Li , Y. Sun, J. Alloy Compd., 2009, vol. 467, pp.347–50.

[44]   Z. Zhaohui, W. Fuchi, Inter. J. Refractory Metals and Hard Mater., 19 (2001) 177-182

[45]   Kyong H. Lee , Seung I. Cha, Ho J. Ryub, Morris F. Dilmore , Soon H. Honga, J. Alloy Compd., 2007, vol. 434–35, pp. 433–36

[46]   C.H. Caceres, D.M. Rovera, J. Light Metals., 2001, vol.1, pp. 151–56.

[47]   C.H. Caceres, A. Blake, Phys. Status Solidi A., 2002, vol. 194, pp.147–58

[48]   J.H. Schneibel, C.T. Liu, M.K. Miller, M.J. Mills, P.M. Sarosi, M. Heilmaier, D. Sturm, Scripta Mater. 2009, vol. 61, pp. 793-96.

[49]   J.H. Schneibel, M. Heilmaier, W. Blum, G. Hasemann, T. Shanmugasundaram, Acta Mater. 2011, vol. 59, pp. 1300-08.

TABLE CAPTIONS

Table 1:   Chemical composition of the powder blends subjected to mechanical alloying

Table 2:   Surface area (from BET analysis) and true density of the mechanical alloyed powders (alloy A, B, C and D) after 40 h milling

Table 3:   Summary of EDS analysis of all the four alloys in mechanically alloyed (40 h) and sintered conditions

Table 4:   Summary of mechanical properties of alloys A, B, C and D as a function of high pressure sintering temperatures

Table 5:   Comparison of compressive stress of alloys A, B, C and D consolidated at 1273 K (1000 °C) by high pressure sintering (HPS), hot isostatic pressing (HIP) and pulse plasma sintering (PPS) processing techniques.