



OPTIMIZATION OF SYNTHESIS OF ODS 9CR MARTENSITIC STEEL BY MECHANICAL ALLOYING

T. S. Kavithaa¹, R. Subramanian¹, P.C. Angelo¹ & P. Shankar²

¹Department of Metallurgical Engineering, PSG College of Technology, Coimbatore

²Surface Science Division, IGCAR, Kalpakkam

ABSTRACT

The present study aims at the optimization of small scale synthesis and bulk production of ODS 9Cr martensitic steel alloy powders by Mechanical Alloying (MA) of elemental metal precursors. Characterization of MA powders produced in a planetary ball mill, an attritor and a double cone blender employing XRD and SEM are reported in detail. The effect of the various process parameters involved in the production of ODS alloy powders are also described. SEM images depict the various stages during the process of MA and XRD studies have confirmed the formation of solid solution.

Keywords ODS – Oxide dispersion strengthening, MA – Mechanical Alloying, EDAX – Energy Dispersive Analysis of X-rays

1. INTRODUCTION

Advanced nuclear energy (Generation IV) systems aim to provide significant improvements in economics, safety, proliferation resistance and sustainability. To achieve these advancements, these systems have to be operated at much higher temperatures and in higher radiation fields requiring the use of materials with adequate high-temperature strength, creep properties, physical properties and metallurgical stability at the operating temperature. [1]

Proper choices of cladding and structural materials are essential for the safe and reliable operation of any Generation IV system. [2] Austenitic stainless steels of type 316 or type 304 were originally the reference core component alloys in FBR plants. [3] Extensive studies carried out to develop the optimum combination of properties for steels intended for use as wrappers [4] indicated that 9-12Cr type ferritic steels presented superior high temperature strength at temperatures below 600 °C [5-7] and excellent dimensional stability at high displacement doses. [3, 5, 8, 9] 9Cr-1Mo (T9) type of steel was initially developed in the 1930s for use in petrochemical and chemical plants, gas turbine engineering, aircraft and aerospace industries, electrical power plants and as nuclear fission and fusion reactor components. Modified 9Cr-1Mo steel (T/P/F 91) with optimized V and Nb contents with high yield and creep-rupture

strengths as well as allowable stresses is now being increasingly used for advanced power plants. [10]

Development of low or reduced-activation materials with enhanced radioactive decay characteristics which has focused on the issue of radioactive waste disposal or recycling of materials from fusion power plant components after they have reached the end of their service lifetime with objectives to maximize the safety advantages of fusion, enable material as well as component maintenance, waste management, and recycling. [9] The development of Ferritic-Martensitic 9-12% Cr steels with Reduced Activation (RAFM steels) for nuclear fusion is based on the excellent irradiation performance of commercial 9-12% Cr steels under high neutron dose exposure in Fast Breeder Reactors[11, 12] and the possibility to achieve a reduced neutron-induced long-term activation alloy by appropriate modification of major alloying elements. [13]

In the development of low activation steels, the international fusion materials programs have narrowed the focus to steels having Cr in the range 7-9% [14], 1-2% W and V and Ta as carbide forming elements modified for reduced activation properties and for improved resistance to hardening-induced shifts in fracture properties. [11, 13, 15] The main drawback of RAFM steels is the limitation of the maximum operating temperature to 550°C. Continuous efforts have been carried out over the years to increase the operating temperature window of existing structural materials of nuclear power plants by solution

Corresponding author: kavithaa.subramanian@gmail.com

strengthening or by precipitation hardening of the alloys. However, excessive amounts of alloying additions result in alloys which could not be worked while precipitates in precipitation hardening alloys become over aged at higher temperatures. To overcome these limitations, ODS (Oxide Dispersion Strengthening) of alloys in which very tiny oxide particles of alumina, thoria, titania and yttria on a submicron scale are dispersed [16] by mechanically alloying to improve the creep strength in addition to providing enhanced radiation resistance, thereby providing a pathway for near-term commercial utilization of advanced new materials. [14, 17]

ODS ferritic/martensitic or ferritic steels offer the potential for revolutionary improvements in high temperature performance, corrosion and radiation resistance, highly resistant to thermal recovery of the material structure, meet low activation criteria, have the potential to significantly advance the performance of components for all the primary Generation IV concepts. [14, 17-26]. The present study aims at the indigenous synthesis and development of a 9Cr-ODS Reduced Activation Martensitic steel by MA.

2. EXPERIMENTAL METHODOLOGY

The present work is on the optimization of small scale synthesis and bulk production of ODS 9Cr alloy powders and was accomplished by MA of elemental metal powders constituting the following chemical composition, in weight percent Fe-9.2Cr-2W-0.204Ti-0.28V-0.01Mn-0.01Si-0.152C-0.02Ni-0.008Mo-0.33 Y₂O₃ employing a Retsch Planetary Ball Mill, an Attritor and a Double cone blender. The ball to charge ratio was maintained as 10:1 while 1wt% stearic acid was utilized as the process control agent in all the experiments. X-ray Diffraction (XRD) analysis of the alloy powders milled for various durations employing the above mentioned routes was carried out using Shimadzu XRD 6000 unit to ensure the formation of solid solution phase. Synthesis of NC yttria used as the dispersoid was prepared through an economic, eco-friendly and efficient Sol-gel method reported earlier by the authors. [27]

Initially trial experiments were taken up in a Retsch Planetary Ball Mill (PM400). Using high carbon steel balls of 10mm diameter as the grinding media, the metal precursors were milled for various milling durations (30-50h) at two different milling speeds (200 & 300 rpm). 200g of powder charge could be milled in a single run of the planetary ball mill. The dispersoid yttria was added during the final 5h of milling of the metal powders in most of the experiments, except in a few cases. In order to study

the effect of addition of yttria on milling trial synthesis of ODS alloy powders were taken up wherein the dispersoid was added at the beginning itself. Mechanically alloyed powder samples, without the addition of yttria, were also prepared and taken up for characterization for comparison. Alloy powder synthesis was also carried out employing stainless steel cylinders as the grinding media to study the effect of morphology of the grinding medium on the efficacy of the milling process.

Alloy powders were also synthesized in a locally designed and fabricated attritor mill of 0.75 litre capacity capable of producing 200g of alloy powders. The attritor was operated at 500rpm (clockwise rotation). Bearing steel balls of 6mm diameter were chosen as the grinding media. The individual metal powder charge of the above said composition was thoroughly mixed and milled for 10 – 75h. Alloy powder samples at milling intervals of 10h were taken up for characterization studies employing XRD and Scanning Electron Microscopy (SEM) to investigate the effect of attritor milling.

Alloy powder synthesis was also taken up in a locally fabricated Double cone blender of 2 litre capacity wherein 250-300g of powder charge could be accommodated and milled. The blender was operated at 40rpm for various durations of milling (0-25h) employing 6mm diameter bearing steel balls as the grinding media and alloy powder samples were taken at intervals of 5h for characterization studies.

3. RESULTS AND DISCUSSION

3.1 Alloy powder synthesized in a Planetary Ball Mill

The results of XRD analysis of alloy powders milled employing a planetary ball mill for various durations at 200 and 300 rpm are tabulated in Tables 1 and 2 respectively. The alloy powder specimens were analyzed in the XRD unit, scanned from 10-80° at a scan speed of 5°/minute using Cu-K α radiation. Table 1 indicates the presence of W peaks even after 40h of milling at 200rpm. Formation of a single phase solid solution of Fe was evident after 45h of milling at 200rpm. Presence of yttrium peaks after 45h of milling suggests the possibility of dissociation of yttria due to the presence of excess energy within the system. The observed peaks were of less intensity due to the low volume fraction of yttria. Addition of yttria at the commencement of milling indicated a negative effect on the milling process due to the presence of W peaks even after 56h of milling at 200rpm. Grain size calculations employing the Debye-Scherrer formula,

$d=0.94\lambda/B \cos \theta$ where B is the FWHM of the peak, λ the wavelength of the radiation and θ the incident angle of the radiation was estimated to be about 10-12nm after 50h of milling at 200rpm.

Table 1 Consolidated XRD data of planetary ball milled powders at 200 rpm

Milling duration	2θ deg	Observed Phase
As-mixed metal precursors (0h)	44.6533	Fe/Fe ₂ Ti/ Fe ₇ W ₆ /Cr ₂ Nb
	40.24	Cr ₂ Nb/W
	64.9816	Cr
25h	44.7311	Fe/Cr
	64.61441	Fe/Cr
30h	40.30573	Ti/W
	44.Aa86114	Fe/Cr
	64.7010	Fe/Cr
35h	40.43533	Ti/W
	44.77717	Fe/Cr
	64.69153	Fe/Cr
40h	40.35389	Ti/W
	44.90601	Fe/Cr
	64.88419	Fe/Cr
45h	40.57424	Ti/W
	44.85056	Fe/Cr
	64.63454	Fe/Cr
50h	65.06204	Y
	44.50753	Fe/Cr
	64.65394	Fe/Cr
50hY	44.55783	Fe/Cr
	65.29920	Y
51h	44.8260	Fe/Cr
	65.14988	Y
56h	44.85351	Fe/Cr
	64.92638	Fe/Cr
	40.38182	W
75h	44.61745	Fe/Cr
	64.66371	Fe/Cr
	40.21463	W

XRD of the alloy powders milled at 300rpm indicated that all the elements go into solid solution of the ferrite phase after 10h of planetary ball milling. Hence 15-20h of planetary ball milling at 300rpm was found to be effective for complete alloying of the metal precursors. Excessive milling resulted in the formation of compounds rich in chromium.

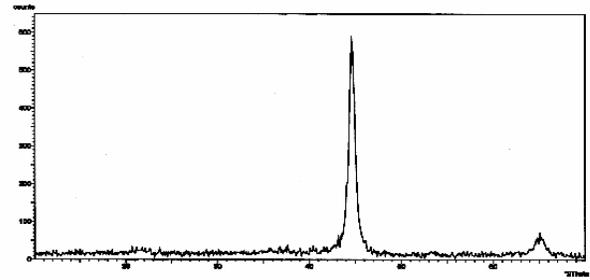


Fig.1 XRD of 50h (200rpm) alloy powder

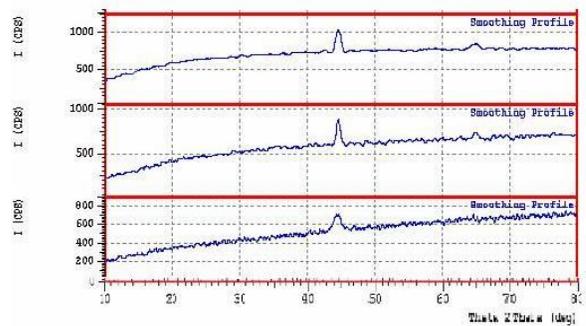


Fig. 2 XRD of 10h, 15h, 20h (300rpm) alloy powder

This could be interpreted as possible oxidation of alloy powders resulting in the formation of chromium oxide which in turn yields compounds rich in chromium. Grain size estimations reveal the average grain sizes of alloy powders milled at 300rpm for 15-20h as 8-12nm. Absence of impurity peaks reveal the purity of the alloy powders synthesized employing the planetary ball mill.

XRD analysis of planetary ball milled (300rpm) alloy powders with stainless steel cylinders as the grinding medium indicated (Table 3) chromium pick-up from stainless steel cylinders by the powder charge which was evident from the physical deformation of the cylinders after completion of milling. Presence of Ti and Cr peaks after 15h and 20h of milling respectively indicated incomplete alloying of the metal precursors into Fe. This could result in increase in milling duration required for ensuring the formation of the solid solution phase. Hence it can be concluded that stainless steel cylinders as grinding media are not preferred to steel balls though it provides higher contact surface area between the grinding media and the powder charge, a primary requirement for efficient alloying of the metal powder charge.

Table 2 Consolidated XRD data of planetary ball milled powders at 300 rpm

Milling duration	2θ deg	Observed Phase
5h	44.5500	Fe/Cr
	64.7450	Cr
10h	44.6266	Fe ₁₇ Y ₂
	64.7800	YS
15h	44.6133	Fe/Cr/Fe ₂ Nb/Fe ₂ Ti/ Fe ₄ Y/Fe ₂ W
	64.9300	Fe ₁₇ Y ₂
20h	44.4200	Fe
	64.6350	YS _x
	67.1050	Fe ₄ Y
25h	44.5266	Fe ₂ Ti/Fe ₂ Nb/Fe/ Fe ₇ W ₆ /Cr ₂ Nb
	45.1400	Cr ₂ Ti
	67.6700	Ti/Fe ₇ Nb ₆
35h	44.6600	FeCr/WCrTi
	43.9400	Fe/Fe ₄ Y/Fe ₁₇ Y ₂ /FeNb /Fe ₇ Nb ₆
	41.5816	FeNb
40h	44.5750	Cr ₂ Nb/Fe ₂ Ti
	58.4666	Fe ₄ Y/Fe _{15.4} V _{1.53} Y ₂
	59.2733	Fe ₄ Y
45h	44.5200	Fe ₇ W ₆ /Fe ₂ Ti/Cr ₂ Ti/ Cr ₂ Nb/Fe
	43.6800	Fe ₇ W ₆ /Fe _{0.2} Nb _{0.8} / Cr ₂ Nb
	64.9775	Cr

Table 3 XRD data of alloy powders milled in a planetary ball mill with stainless steel cylinders as the grinding media at 300 rpm

Milling duration	2θ deg	Observed Phase
15h	44.5516	Fe ₇ W ₆ /Fe ₂ Ti/CrNb ₆ /Fe/Fe ₁₇ Y ₂
	64.7100	Fe ₂ W
	40.2300	Ti/Cr ₂ Nb/Cr ₂ Ti/Fe ₇ Nb ₆ /Cr ₂ Nb
20h	44.5250	Cr
	64.6300	Cr, YS _x

3.2 Alloy powder synthesis employing an Attritor Mill

In case of milling of the powder charge in the attritor the dissociation of the ceramic dispersoid yttria occurred after 10h of milling which was evident from the peaks of compound of iron and yttrium. Addition

of the yttria into the system via attritor milling is not recommended for attaining a fine distribution of the dispersoid in the matrix.

Table 4 Consolidated XRD data of alloy powders milled in an attritor at 500 rpm

Milling duration	2θ deg	Observed Phase
As-mixed metal precursors(0h)	44.6533	Fe/Fe ₂ Ti/Fe ₇ W ₆ /Cr ₂ Nb
	40.24	Cr ₂ Nb/W
	64.9816	Cr
10h	44.6350	Fe ₁₇ Y ₂ /Fe
	29.2800	Y ₂ O ₃
	42.6750	Fe ₁₇ Y ₂
20h	44.6733	Fe ₄ Y/Fe ₇ W ₆ /Fe ₂ Ti/Fe
	64.9633	FeYTi/Fe ₁₆ TiY ₂
	79.3033	Fe ₁₇ Y ₂ /FeCr
30h	44.6100	Fe/Fe ₁₇ Y ₂ /Fe ₂ Ti/Fe ₄ Y
	64.7300	Cr
	72.5400	Y
40h	44.6050	Fe ₂ Ti/Cr ₂ Nb
	64.7100	Cr
	79.9600	Fe ₁₇ Y ₂
50h	44.4900	Fe ₇ C ₃ /(CrFe) ₇ C ₃ /CrC
	64.6600	Fe ₁₆ TiY ₂
	72.8300	Y
60h	44.4550	Cr ₂ Nb/Cr ₂ Ti
	39.8800	W
	46.7100	Y ₂ O ₃
70h	44.5250	Ti ₂ C _{0.06} /CrFe ₇ C ₃ /Fe ₇ W ₆
	64.7900	Fe ₁₇ Y ₂ /Cr/Fe/Y ₂ O ₃
75h	44.5000	(CrFe) ₇ C ₃ /TiO ₅
	40.4400	Fe ₁₇ Y ₂ /Ti
	42.8000	Fe ₁₇ Y ₂ /Y

Presence of W peaks after 60h of milling indicated incomplete alloying of the metal precursors in iron. It can be concluded that uniform alloying of the metallic constituents into the Fe matrix could not be achieved in case of MA of the powder charge in an attritor. A reduction in milling speed (rpm) is therefore recommended. A decrement in grain size of the powder charge was observed from 10h – 50h milling in an attritor with a subsequent increase in grain size after 60h of milling. Average grain size of the alloy powders after 50h of milling in an attritor was calculated to be about 9nm.

Table 5 Consolidated XRD data of alloy powders milled in a double cone blender operated at 40rpm

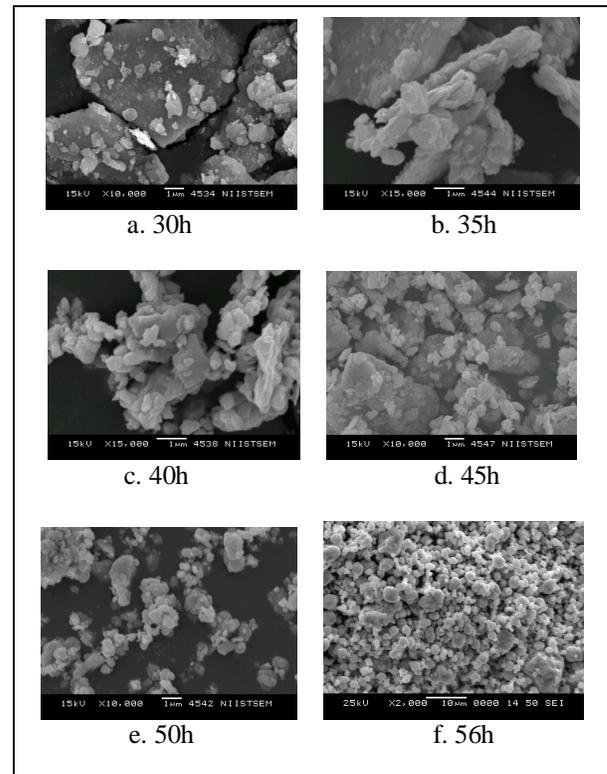
Milling duration	2θ deg	Observed Phase
5h	44.5850	Fe/Cr ₂ Nb
	64.8200	NbTi ₄
	40.2000	Ti
10h	44.6016	Ti ₂ C _{0.06} /Nb ₂ O ₅
	10.6800	Fe ₂ Nb
	51.2100	Ti
15h	44.5866	FeNb/Fe ₇ Nb ₆ /Fe ₂ Nb/Fe ₃ O ₄ /TiC
	64.7800	Nb ₂ O ₅ /YS _x /NbO ₂ /W C ₃
	51.0650	Fe ₃ O ₄ /YC ₂
20h	44.5266	Fe ₄ Y/Fe ₁₇ Y ₂ /Fe/Fe ₂ Nb/Fe ₇ W ₆
	64.7250	YS _x /Fe ₁₆ TiY ₂
	59.0700	YS _x
25h	44.5450	Fe ₁₇ Y ₂ /Fe ₂ Ti/Fe
	64.7500	Fe ₁₆ TiY ₂
	23.8600	Fe ₂ Ti

3.3 ODS alloy powder synthesis in a Double Cone Blender

X-ray diffraction analysis of the alloy powders milled in a double cone blender indicated that all individual metallic constituents had gone into solid solution of the ferrite phase only after 20h of blending of the powder charge with 6mm bearing steel balls. The above was evident from the presence of Ti after 10h of blending and the formation of WC₃ after 15h of blending. The dissociation of yttrium oxide could be manifested from the formation of compound of iron and yttrium after 20h of blending. Contamination of the powder charge due to pick up of carbon and oxygen from the atmosphere was apparent from the results reported. Further blending (25h) of the alloy powders resulted in the formation of compounds of iron, titanium and yttrium. The average grain size of the ODS alloy powders milled in a double cone blender was estimated to be 9-12nm.

3.4 SEM characterization of ODS alloy powders

SEM of as-mixed metal precursors showed the presence of a mixture of particles most of them in the size range of 50μm with rod shaped morphology and some particles of 10μm size with irregular size and shape. SEM of the ODS alloy powder samples reveal the cold welding of the metal powders after 30h of milling.

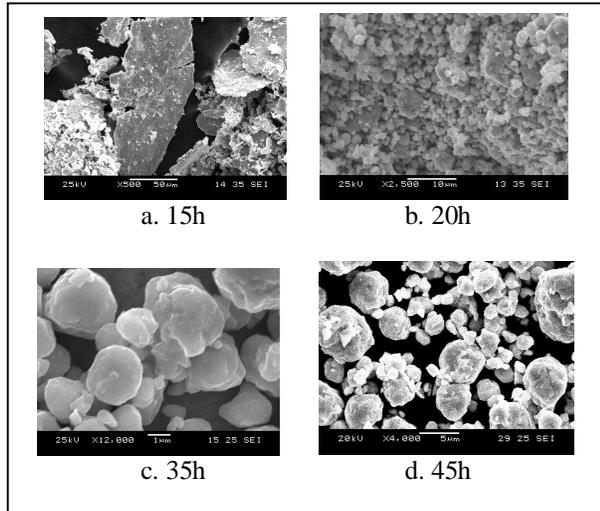
Fig. 3 SEM micrographs of milled powders at 200rpm

The 10μm size particles were embedded with smaller powder particles in the size range of 1μm. Flattening and deformation of the metal powders was observed around 35h of milling wherein most of the particles were 7μm in size (fig. 3b) finely cold welded with smaller particles (<1μm). After 40h of milling, particles break up to sizes <3μm as is evident from the SEM micrographs. Lamellae formation was also observed as shown in figure 3c. Fine agglomerates of powder particles were seen after 45h of milling with a bimodal size distribution wherein most of the particles were in the size range of 1μm together with larger particles in the size range of 3-6μm. 50h of milled ODS alloy powders revealed uniform finer particle size distribution (<1μm). The SEM micrograph of alloy sample milled for 56h showed narrow size distribution of 1-4μm particles with few particles in the size range of 7-10μm.

SEM micrographs of the ODS alloy powders milled at 300rpm revealed the various stages of MA. The progressive nature of alloying is evident from figures 4 (a-d). 20h milled ODS alloy powder revealed finer size distribution of particles in the range of 1-2μm

which reinforces the importance of increase in milling speed.

Fig. 4 SEM micrographs of milled powders at 300rpm



Extensive milling of the powders resulted in the formation of agglomerates (3-4 μ m) as shown in fig. 4d. SEM analysis of ODS alloy powders milled employing a blender is shown in figure 5. The powder particles had irregular size and shape after 5h of blending. Flattening of the powder charge and regularity in the morphology of the particles were observed after blending for 10h. Cold welding was found to occur around 15h which was followed by subsequent particle break up after completion of 20h of blending. Most of the particles were in the size range of 10 μ m and some of the particles had sizes in the range of 50 μ m and 1-3 μ m. Further blending aided re-welding of the powder charge thereby resulting in the formation of substantially uniform size distribution of the particles.

SEM micrographs (figure 6) of alloy powders milled in an attritor (10h) indicated particle break up to sizes of 50 μ m and 25 μ m. After 20h of milling flattening of the powder charge was observed together with further break up of particles to 10 μ m. Cold welding of the powder particles is also evident from the micrographs. 30h of milling resulted in the formation of lamellae and their subsequent agglomeration. Sustained milling in the attritor showed formation of agglomerates of size 100 μ m (40h), their break up to sizes of 50 μ m (50h) followed by formation of finer agglomerates of size 25 μ m (60h & 70h).

Fig. 5 SEM micrographs of powders milled in a double cone blender at 40rpm

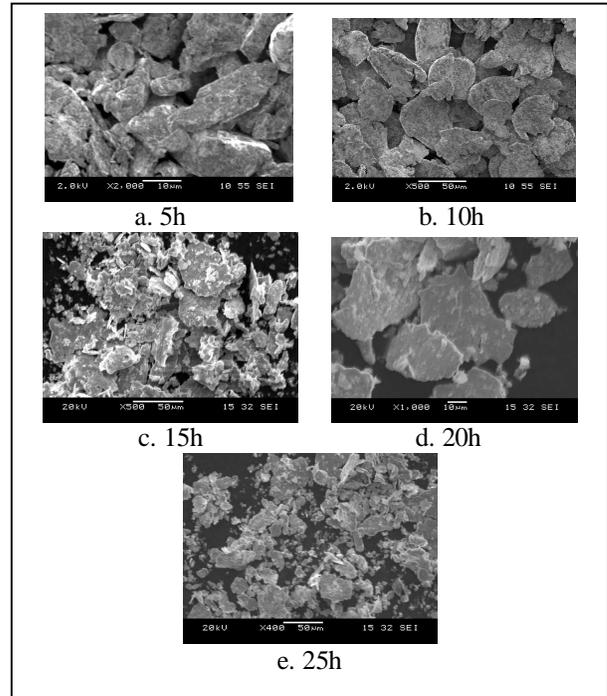
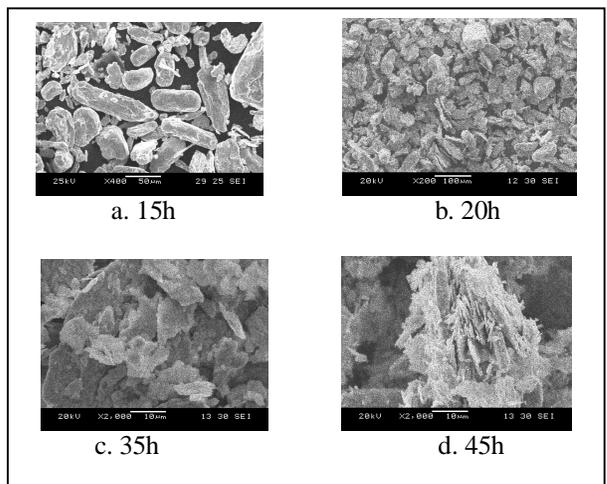


Fig. 6 SEM micrographs of powders milled in an attritor mill at 500rpm



4. CONCLUSIONS

Successful synthesis of ODS Fe-9Cr ferritic steel via MA employing a Retsch Planetary ball mill and a double cone blender has been accomplished and the optimization of the process parameters involved is reported. Though the removal of the powders from the container of the planetary ball mill and cleaning of the container are tedious, the net wastage involved and contamination is lesser. Since efficiency is higher in

the case of planetary ball milling, it is preferred over other equipment for small scale synthesis of ODS alloy powders. The duration required for the formation of solid solution phase was higher for an attritor when compared to milling in a planetary ball mill/double cone blender. SEM characterization studies reveal the progressive nature of alloying namely, particle deformation, fracture and cold welding. Based on the above results, it can be concluded that 15-20h of planetary ball milling at 300rpm has been identified as the optimum process parameter for efficient small scale production of ODS alloy powders. It can be concluded from the present study that a double cone blender is the most suitable equipment for bulk preparation and production of ODS alloy powder powders.

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