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Elaboration and study of mechanically alloyed O.D.S. ferritic stainless steel

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ABSTRACT

O.D.S. ferritic stainless steel has been elaborated by mechanical alloying from elemental powders and fully characterized by SEM, TEM, EDS and XRD.

Macroscopic and microstructural evolutions of the material have been determined as a function of milling time. It has been observed that the changes in composition and structure are dramatic up to 40 milling hours. For higher milling durations, the powder characteristics are nearly unchanged.

Two kinds of reinforcing dispersoids have been identified by TEM on HIP compacted powders: titanium compounds (50-200 nm) in the grain boundaries and complex yttrium oxide nanometer sized particles inside the grains.

1. INTRODUCTION

Mechanical alloying, a powder metallurgy technique, can be used to produce metal matrix materials reinforced by ultrafine ceramic dispersions [1,2,3]. In this technique, metallic powders are repeatedly welded, fractured and rewelded in a high energy mill, giving birth to new particles with a characteristic layered structure [4]. Because of numerous shocks with steel balls, the layers become thinner and thinner until they can no longer be optically resolved. At that time, a correct degree of mechanical alloying is achieved. Milling with additional oxide particles introduce nanometer dispersoids between metallic layers resulting in an Oxide Dispersion Strengthened alloy (ODS) in which mechanical properties, mainly creep resistance, are enhanced by pinning of dislocations [5].

Due to their good swelling resistance, ferritic stainless steels can be candidates for nuclear fuel canning applications [6,7] provided their creep strength could be improved [8,9]. The aim of our work was to develop the elaboration by mechanical alloying of such an ODS ferritic steel. Optimization of the process has been obtained by studying the effects of milling conditions on macroscopic and microstructural evolutions of the material.

2. EXPERIMENTAL

Starting materials were chemically pure Fe, Cr, Ti, Mo and Y_2O_3 powders whose characteristics are given hereafter:

	Fe	Cr	Ti	Mo	Y_2O_3
Particle size (μm)	100-150	300	<100	4	<44
Weight %	bal.	14	1	0.3	0.25

Mechanical alloying was carried out in a Netzsch PR01 attritor charged with 9.5 kg of 8 mm diameter 100C6 steel balls and 400 g of blended powders. Rotating speed was 400 rev/min. Milling was performed under air and under argon atmosphere from 12 to 100 hours.

Elaborated powders were characterized by optical and electron microscopy. Ultramicrotomy was used to prepare 60 nm thick sections for STEM examinations performed on a JEOL 2000 FX microscope equipped with Energy Dispersive Spectroscopy (EDS) on a TRACOR 5400-B using MSCAN 2 (an X-imaging program). X-ray diffraction (XRD) was used to determine phases transformations within the powders. Oxide dispersion was observed on carbon replicas of consolidated powders after hot isostatic pressing (HIP).

3. CHARACTERIZATION OF MILLED POWDERS

3.1. Morphology

SEM photographs show that powder particles become considerably smaller with increasing milling time under air (fig.1). After a few hours, the powders consist of flaky particles with a mean diameter of 100-150 μm . Afterwards, the particles become nearly spherical and their diameter decreases to approximatively 20-50 μm . Further milling reduces the particles size down to a few micrometers (fig.2). At this size, the particles tend to agglomerate.

Fig.1 : Evolution of powders morphology as a function of milling time under air (a: 12 h, b: 20 h, c: 40 h, d: 60 h)

Fig.3 shows powder grain microstructures on TEM samples prepared by ultramicrotomy. For 20 hours milling (fig.3a), the powders are constituted of parallel layers of about 50-100 nm. It has been observed by electronic microprobe that these layers correspond to pure elements lamellae. After 80 hours milling, microstructure is optically homogeneous (fig.3b).

Fig.2 : Macroscopic powder characteristics versus milling time

Fig. 3 : TEM photographs of ultramicrotome sections of powder grain after milling under air (a: 20 h, b: 80 h)

3.2. Composition

Chemical analysis has confirmed that the global composition of the milled powder corresponds to the expected one. STEM-EDS allowed determination of the local compositional homogeneity of an ultramicrotome cut. The compositional difference (%) between measured and nominal Cr/Fe ratio was determined locally for different milling times. Results are given in fig.2

Because of the similarity in Fe and Cr cristallographic structure, the superimposition of their diffraction peaks and their absorption coefficients, the monitoring of phases transformations with milling time by XRD analysis was uneasy. Fe, Cr, and FeCr peaks were separated using a deconvolution program. The peak intensity was observed to decrease with simultaneous broadening and shifting. After 40 milling hours, Fe and Cr could no longer be separated, and only one phase could be identified as FeCr solid solution [10].

3.3. Microstructure

The broadening of XRD peaks with increasing milling time can be understood due to both strain and crystallite size reduction. This refinement of matrix microstructure has been confirmed by dark field TEM examinations (fig.4a). At the first stage of milling, mean crystallographic grain size is about 100 nm. After continued milling (fig.4b), the final grain size becomes ten times smaller (fig.5). The transition between crystallized and microcrystallized structure is evidenced by diffraction patterns of fig.6.

Fig.4 : Dark field TEM photographs of milled powders (a: 20 h, b: 40 h)

Fig.5 : Microscopic powder characteristics versus milling time

The results obtained from the different characterization techniques show that 40 hours is an optimum time for milling under air:

- the powder grain and crystallite size remain relatively constant after 40 hours,
- the chemical composition is nearly homogeneous and pure Fe and Cr phases are no longer observable by XRD. Hot consolidation (extrusion, rolling, HIP) of the powders will smooth any residual compositional heterogeneities.

Some experiments performed under an argon atmosphere have shown that, in this case, chemical homogeneity and crystallite size are identical for 40 hours milling. Otherwise, powder grains are elongated and ten times bigger.

Fig.6 : Diffraction patterns of milled powders (a: 20 h, b: 40 h; c: 100 h)

4. CHARACTERIZATION OF CONSOLIDATED MATERIALS

After milling 40 hours under argon atmosphere, powders were canned and compacted 3 hours at 920°C. TEM samples were prepared by conventional methods.

Submicronic matrix structure was generally observed although several microns grains regions could be found. Using the carbon replicas technique, two kinds of dispersoids were observed in the densified material. The biggest ones (50-200 nm), essentially aligned in some of the grain boundaries, have been identified by electronic diffraction as Ti (C,N) compounds (fig.7). The distance between these alignments corresponds approximatively to as-milled powders dimensions. These compounds can result from powder grains superficial contamination when handling under air before canning. The second dispersion, too small to allow electronic diffraction (10-20 nm), was analysed by energy dispersive spectroscopy (EDS) as complex γ , Ti, Fe oxides (fig.8).

Fig.7 : TEM photographs of Ti (C, N) dispersoids

Fig.8 :TEM photographs of (10-20) nm Y , Ti , Fe oxides

5. CONCLUSION

Fe-Cr-Ti-Mo ferritic steel reinforced by yttrium oxide dispersion has been elaborated from elemental powders by mechanical alloying in an attritor mill.

Macroscopic and microstructural evolutions of the material have been studied by SEM, TEM, EDS and XRD for milling times ranging from 12 to 100 hours:

- after short milling time, powders are composed of 50-200 μm elongated grains showing a lamellar structure corresponding to the stacking of elemental Fe and Cr layers,
- after milling time, particles are nearly equiaxed with a granulometry of 5-10 μm . Material composition is homogeneous at a very fine scale. Crystallite size is reduced to 20 nm.

It has been demonstrated that 40 hours is an optimum milling duration after which material compositional and structural changes are small.

Two kinds of reinforcing particles have been observed by TEM on HIP compacted powders:

- Y_2O_3 ultrafine particles (10-20 nm) associated with Ti and Fe are dispersed inside the grains,
- 50-200 nm Ti (C,N) compounds are aligned in the grain boundaries.

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