Alternative fabrication routes towards oxide dispersion strengthened steels and model alloys

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Abstract. The standard powder metallurgy (PM) route for the fabrication of ODS steels involves the gas atomization to produce a pre-alloyed powder, mechanical alloying (MA) with fine oxide powders, consolidation, and finally thermal/thermo-mechanical treatment. It is well-established that ODS steels with superior property combinations, for example creep and tensile strength, can be produced by this PM/MA route. However, the fabrication process is complex and expensive, and the fitness for scaling up to the industrial scale is limited. At the laboratory scale, production of small amounts of well-controlled model systems continues to be desirable for specific purposes, such as modelling-oriented experiments. Thus, from the laboratory to industrial application, there is growing interest in complementary and/or alternative fabrication routes for ODS steels and related model systems, which offer a different balance of cost, convenience, properties and scalability. This paper reviews the state of the art in ODS alloy fabrication and identifies promising new routes towards ODS steels. The PM/AM route for the fabrication of ODS steels is also described as it is the current default process. Hybrid routes that comprise aspects of both the PM route and more radical liquid metal (LM) routes are suggested to be promising approaches for larger volumes and higher throughput of fabricated material. Although similar uniformity and refinement of the critical nanometer-sized oxide particles has not been yet demonstrated, ongoing innovations in the LM route are described, along with recent encouraging preliminary results for both extrinsic nano-oxide additions and intrinsic nano-oxide formation in variants of the LM route. Finally, physico-chemical methods such as ion beam synthesis are shown to offer interesting perspectives for the fabrication of

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model systems. As well as literature sources, examples of progress in the authors' groups are also highlighted.

I. INTRODUCTION

The development of oxide dispersion strengthened (ODS) steels reaches back to the 1960s[1] (see also [2,3] and references therein) and has been continuing since.[4-6] ODS steels have been shown to offer improved creep strength over their non-strengthened equivalents, extending the potential application window of conventional high-Cr steels from less than 873 K (600 °C)[2,7] towards 1073 K (800 °C),[8] while maintaining other favorable properties of high-Cr steels, in particular in civil nuclear power environments. It is interesting to note that while powder metallurgy (PM) fabrication routes including milling and consolidation of steel/oxide powder blends, or controlled oxidation of Y-alloyed steel powders were considered from the very beginning[1,2,9], the notion of mechanical alloying (MA) did not make a significant appearance in the literature until 1970.[10]

After a period of research and development commercial ODS products became available in the 1990s, and two lines of scientific effort received special interest. First, composition and processing parameters were varied to obtain optimum microstructures and/or optimum mechanical properties. For instance, in a key finding it was shown that the addition of approximately 0.4 wt% Ti to a master alloy gave rise to a reduction in the oxide-particle size from approximately 20 nm to about 2 nm[5] and an improvement in creep resistance. Consolidation techniques for MA powders include hot-isostatic pressing (HIP), hot extrusion (HE) and, more recently, pulsed-current-assisted sintering also known as spark plasma sintering (SPS) or field assisted sintering technique (FAST). The role of post-processing of consolidated products including thermo-mechanical treatment has also been investigated extensively. Second, significant effort was expended by the desire to simplify the overall process, to make it less expensive, more reproducible, and to allow scaling up towards the industrial scale to be achieved, even where some compromise in the performance of the material might be needed. However and as discussed later, it remains an open question as to what quantified curtailment of PM/MA ODS alloy (a few kg) properties could be tolerated in order to realize ODS alloys at the industrial scale (100's to 1000's kg).

In the last few years the number of approaches applied to fabricate ODS steels or dispersion-hardened model alloys has increased. A comprehensive source of information on these efforts are international workshops such as the Workshop on Fe-based ODS alloys: Role and future applications (University of California at San Diego, California, November 17-18, 2010[11]), the International Workshop on Dispersion Strengthened Steels for Advanced Nuclear Applications (DIANA, selected talks published in vol. 428 of Journal of Nuclear Materials), and special sessions of general and nuclear materials conferences such as the International Conference on Fusion Reactor Materials (ICFRM). Due to the wide variability of reported and possible alternative approaches

towards ODS steels, national and international collaboration is vital for significant progress to be achieved. The Joint Program on Nuclear Materials (JPNM) of the European Energy Research Alliance (EERA) represents a suitable umbrella for such type of international collaboration. An EERA JPNM Pilot Project on "Alternative fabrication routes towards ODS steels" (AFROS) has recently been launched. The majority of the co-authors of this study are involved in the AFROS project.

The first objective of the present study is to summarize and categorize broadly the approaches to ODS steel fabrication from the published literature in order to simplify orientation in the field. In doing so, broadness of the review is preferred over in-depth analysis of individual reports. Four major groups of approaches are distinguished: the default PM/MA route, liquid metal (LM) routes, hybrid routes based on elements from both the PM and LM route, and physico-chemical methods that are focused on the fabrication of model alloys.

The second objective is to highlight selected original results recently obtained by the co-authors' groups in relation to the AFROS project introduced above, with more in-depth descriptions published subsequently elsewhere. The purpose of including them here is to provide the very latest information on work-in-progress, which may be of use to similar groups. Some specific highlights include SPS approaches to reduce sintering time and limiting coarsening of the microstructure, controlled oxidation of Y-alloyed gas atomized powder, Y-alloyed melt-spun ribbons and spray formed billets as an approach to avoid the MA step, and contactless alternating-magnetic-field-induced cavitation as a method to introduce nano-oxides directly into traditional LM routes.

II.   FABRICATION ROUTES TOWARDS ODS STEELS AND MODEL ALLOYS

A. *The standard powder metallurgy/mechanical alloying (PM/MA) route for ODS alloys*

The standard PM/MA route for the fabrication of ODS steels consists of the following major steps:[5]

- selection of the steel composition,
- gas atomization of the steel to be dispersion hardened (master alloy),
- blending of the steel powder and suitable oxide powders, e.g. commercially available 20-200nm Y_2O_3,
- mechanical alloying of the blended mixture using e.g. planetary ball mills or attritor mills,
- canning and degassing,
- consolidation, e.g. by means of HIP or hot extrusion (HE),
- post-processing based on thermal, mechanical or thermo-mechanical treatment (TMT).

Since the basics of this were established more than 40 years ago, there has been steady progress in each of these steps, and some notable advances are described below. For the purpose of the identification of promising alternative fabrication routes, the standard PM/MA route will serve as a reference. A comparison of some experimental and commercial alloys was reported by Klueh *et al.*[7] from the viewpoint of nuclear applications. A set of optimal processing conditions was reported by Baluc *et al.*[12] for Fe-14Cr-2W-0.3Ti-0.3Y_2O_3 ODS reduced activation ferritic steel produced from elemental powders by means of PM/MA and HIP.

Several types of alternative approaches indicated in Figure 1 will be considered in some more detail below.

B. *Recent progress in powder processing of ODS alloys*

From the viewpoint of the composition of ODS steels, recent efforts have focused on transformable (e.g. Fe-9%Cr-X) alloys including reduced activation variants such as ODS-Eurofer[13] and ferritic (e.g. Fe-14%Cr-X) alloys. The addition of approximately 0.4 wt% Ti to alloys of the type was a key finding that was crucial in both cases in achieving ultrafine particle dispersions.[5] Cladding tubes, one of the most advanced potential nuclear energy applications, have been produced successfully from both alloy variants.[14] Comprehensive microstructural and mechanical characterization of the tube materials is in progress within the European Project MatISSE (Materials Innovations for Safe and Sustainable Nuclear in Europe). The effect of the type of added oxide powder, including MgO, La_2O_3, Ce_2O_3 and ZrO_2 on the microstructure and mechanical properties of HIPed Fe-13%Cr steel, has been investigated at the laboratory scale and compared with Y_2O_3 additions, which is generally considered the default oxide addition.[15] The MgO and Ce_2O_3 containing alloys performed better than the reference alloys, at least in terms of Charpy impact tests.

Recent progress in gas atomization of liquid metals was achieved using a Laval nozzle.[16] The design and evaluation of a Laval-type supersonic atomizer for low-pressure gas atomization of molten metals was reported by Si *et al.*.[17] The so-called Nanoval process[18] based on a Laval nozzle is utilized in section III.A for steel powder production within own work in progress. This process allows a narrow distribution of spherical powder particles to be achieved on the industrial scale.

In terms of the critical milling stage in which the ceramic oxide particles are dispersed into the alloy matrix, there are various types of mills available including attritor mills, planetary ball mills and shaker mills. Here it is important to note that the introduction of horizontally rotating attritor mills has increased powder throughput significantly.[19] The type of mill, milling atmospheres

(argon, hydrogen) and milling parameters (revolutions per minute, ball to powder ratio, milling time) have been varied individually or in combination in many studies. These influence factors are typically cross-correlated with one another and with other parameters such as steel composition and initial powder quality, and depend on the details of the specific milling equipment used. In other words, *a universal* set of parameters that guarantees optimum properties does not exist.

Although the interaction of the ceramic oxides with alloy powders during milling is critical for the subsequent formation of refined nano-oxide clusters, there is ongoing uncertainty and ambiguity as to the precise mechanism, and whether one or more mechanism may operate under any particular set of milling conditions. For example, compelling evidence has been presented that Y_2O_3 powder particles became ultra-fine and defective during milling so that during X-ray diffraction investigations, they contribute to an amorphous background to the spectra, and can no longer be easily distinguished.[20] Supporting atom probe studies that showed Y and O remained strongly spatially correlated, even at a domain sizes of only a few nanometers[20]. Other work using small angle neutron scattering (SANS) has suggested that alternatively, or possibly in addition, Y_2O_3 may dissociate to Y and O which then dissolve in the highly defective bcc lattice, forming a solid solution with the Fe alloy matrix.[6] However, refinement below the detection limit of X-ray diffraction (XRD) or SANS (~1 nm) may be occurring. Overall, at such small length-scales of nanometer or less, the ability and importance of distinguishing between tiny fragments of amorphous Y_2O_3 and dissociated Y and O is perhaps a moot point: what has been shown is that at these levels of refinement, acceptably uniform distributions of refined oxide-dispersoids (especially in the presence of Ti) can form on subsequent heat treatment. Intensive research efforts are now devoted to understanding the nucleation and/or devitrification, and growth behavior of the nano-oxides, and precisely the role of refining alloy additions such as Ti.

The consolidation techniques of HIP and HE are well-established and effective for ODS-based powders. Nonetheless, more recently, SPS and other field-assisted consolidation techniques have received increasing interest because of their potential to reduce process times and thermal exposure during processing, and thus retain desirable refinement or metastable features of the milled powders into bulk material. For example, SPS has been applied to FeAl intermetallics,[21] Fe,[22] Fe-9%Cr,[23-25] Fe-14%Cr-X,[26-30] and Fe-16%Cr-3%Al.[31] SPS was shown to be suitable for the fabrication of semi-industrial batches of ODS steel (~0.5 kg).[32,33] A typical (but not limited to SPS) observation of the resulting microstructures is a bimodal grain size distribution,[22,30,34,35] which is a typical feature of alloys with significant grain boundary stabilization (Zener pinning) due to fine-scale particles after thermal exposure.[34,36] It is interesting to note that bimodal grain size distributions have been suggested to be a potential basis for favorable strength-ductility combinations of

nanostructured materials in general[37,38] and for ODS steels in particular.[39] Bimodality with respect to creep properties requires specific consideration of the temperature-stress regime, because different creep mechanisms such as diffusion creep, dislocation creep and grain boundary sliding exhibit different dependencies on grain size.

Field-assisted hot pressing (FAHP), using a low-frequency alternating current, was also applied successfully by Garcia-Junceda *et al.*[40] and the effectiveness of microwave sintering over conventional vacuum sintering with respect to refined grain sizes was reported by Yao *et al.* for a 14%Cr ODS steel.[41] Electric pulse consolidation techniques as alternatives to spark plasma sintering were reviewed by Yurlova *et al.*[42] and Bogachev *et al.*[43], confirming its effectiveness for accelerated consolidation of 13%Cr ODS steel powders.

The application of friction consolidation processes to ODS steels was suggested by Catalini *et al.*[44,45] who gave a preliminary demonstration of ODS powder consolidation and ultra-fine oxide precipitation, and was suggested to offer the potential for reduced processing costs.

The role of differing types of thermo-mechanical treatment (TMT) was investigated by Oksiuta *et al.*.[46] Considering HE, hot pressing and hot rolling of an as-HIPed ODS steel, they suggested that each resulted in a different balance of texture, strength and fracture behavior and no single process or combination of TMT parameters was universally preferred.

Finally, severe plastic deformation (SPD) processes such as equal channel angular extrusion (ECAE) or pressing (ECAP) and high-speed hot extrusion (HSHE) have also applied to ODS steels.[12,47,48] Both HSHE and ECAE of the as-HIPed material were shown to give rise to a significant microstructure refinement and extra hardening.[47,48] However, SPD applied to PM ODS steels may not offer a major step forward from an economic point of view.

C. *Liquid metal processing for ODS alloys*

The inherent attractions, in terms of cost and scalability, of simply adding fine oxide powders directly to liquid steels have ensured it has continued to attract attention.[49,50] Indeed, a popular students' examination question related to ODS steels is why not simply add an oxide powder directly to the metallic melt? Correct answers will refer to the limited wettability of the oxide particles, the tendency to agglomerate before introduction and in the melt, buoyancy of the particles, difficulties in penetrating the oxide skin, and the practical difficulties and possible health and safety issues of dealing with significant volumes of nano-particles in the foundry environment.

Despite these challenges, research efforts have continued in research laboratories at small scale. If an internal precipitation-oxidation step after solidification (described later) is to be avoided – which would be highly desirable from a cost and simplicity point of view – then any oxide particles

to be added into the melt, ideally would be less than 20 nm in size since oxides with smaller diameters are generally held to most desirable. In this respect, nano-milling as an initial step has been shown to be more effective in producing sufficiently fine oxide powders than conventional planetary ball milling.[51]

In order to avoid agglomeration of the nm- or μm-sized oxide particles prior to or during the introduction into the melt, the preparation of low-concentration, low-viscosity Y_2O_3 colloidal powder suspensions has been investigated.[52] As part of this work, there was a systematic study of the contact angle of Fe-Cr droplets onto Y_2O_3 substrates, since an intimate and wetted liquid alloy-ceramic interface is essential for adequate dispersion.[52] The generally weak wettability of Y_2O_3 particles with liquid Fe-Cr alloys was improved by the addition of metallic Y to the melt.[53] It was shown that the steel T91 system with addition of Y_2O_3 in combination with metallic Y gives rise to a homogeneously dispersed ODS T91 material.[53]

Any casting technology for ODS steels requires the solution of several complex problems. The particles must be inserted into the melt, they must be dispersed from one another and across the bulk of the melt, and they must not dissolve or react excessively with the liquid alloy. For particle insertion, approaches including filling into cavities in the feedstock metal ingot, full or partial mechanical alloying to pre-coat the particles with the matrix alloy, electrochemical particle co-deposition for a similar effect, as well as various types of intense stirring usually through the surface of the molten metal. Stirring can also be achieved in a contactless manner by magnetic stirring[54], making use of an alternating magnetic field. The application of a magnetically driven tornado-like vortex, caused by a superposition of two different kinds of alternating magnetic fields, for stirring floating particles into a liquid metal has recently been investigated.[55] It was found that the particle agglomeration survived the high velocity flow in the relatively small melt volume of approximately 0.4 dm^3. This situation is expected to change for much larger volumes.

Once nano-particles are incorporated into the melt, strong capillary and inter-particle forces will promote agglomeration into, at least, micro-sized clusters. In order to avoid this, powerful agitation of the melt is needed. For light alloys such as those based on aluminum, ultrasound is typically used especially at the laboratory scale.[56,57] Unlike simple mechanical stirring or shearing, ultrasound induces expanding and collapsing cavitation bubbles that produce powerful shocks and micro-jets that, on a micro-scale, overcome the forces holding together clusters of nano-particles. Ultrasound also induces a macro-scale streaming effect, causing the melt to be stirred and this action helps both to disperse particles, and to feed agglomerates into the cavitation zone, close to the sonotrode. However, for higher melting alloys such as steels, dissolution of the sonotrode itself is a major problem. It can be speculated that a concentrated ODS alloy sonotrode, which gradually dissolved,

might be an effective way to introduce nano-particles. Because of the difficulties of sonotrode dissolution, contactless magnetic excitation of acoustic cavitation in liquid metals based on a superposition of a steady and an alternating magnetic field has been pursued and recently demonstrated at proof of concept scale.[58]

After the particles are successfully dispersed, they must maintain this dispersion through solidification, and a key problem may arise: if the particle has poor wettability with the solid/liquid interface, it will be pushed ahead of the advancing solidification front, becoming more and more concentrated in the shrinking residual liquid, and eventually they are forced together into large agglomerates in an irreversible gelation-like reaction. Even with aggressive subsequent TMT, these agglomerates will not be broken down sufficiently and nano-particles redistributed acceptably.

Thus, key challenges remain for combining extrinsic fine-scale oxide particles with liquid steels as a viable route to ODS alloys, but the apparent simplicity, cost and scalability will continue to attract research effort and stimulate new approaches.

D. *Hybrid routes*

The term hybrid route here refers to fabrication methods for ODS steels that draw on know-how from both the PM and LM route. However, it can be understood in a broader context to contain elements of chemical, physical or metallurgical principles.

As mentioned in the introduction, the idea of controlled oxidation of a Y-containing steel powder was at the very origin of the development of ODS steels.[1,2,9] There is continuing interest in strategies for controlled internal oxidation of steel powders in order to reduce or eliminate the MA step in the standard route.[59] The in-principle viability of internal oxidation has been demonstrated, for example, in the high-throughput process of gas atomization reaction synthesis (GARS) involving Fe-16Cr alloy powders reported by Rieken *et al*.[60] Here post-solidification internal solid-state O exchange reactions between metastable Cr-rich oxides and Y-enriched intermetallic precipitates gave rise to a uniform distribution of nm-sized mixed oxides. However, an ongoing challenge is to provide similar levels of refinement and control of the *in-situ* formed oxides as is commonly practiced in the PM/MA route. In the PM/MA route, it is likely that the enormous defect density (vacancies and dislocations) created in the heavily milled, heavily work-hardened bcc steel powders plays a critical role in the fine-scale nucleation or devitrification of oxides in the very early stages of heat treatment. The extent to which the extreme refinement of oxides to only a few nanometers that is achievable in MA'ed, Ti-containing ODS alloys can be achieved in bulk processed and internally oxidized alloys has not yet been quantified; on the other hand, the critical engineering need for oxide clusters of only 2 to 5 nm, as opposed to more readily achieved 10 to 20 nm sized particles,

has not been demonstrated for all possible applications. Put another way, while smaller oxides are probably always desirable from a mechanical property point of view, they may simply be unachievable at an engineering scale and coarser but nonetheless enhancing oxides in larger material volumes may still be cost-effective for some applications.

Methods such as spray forming[61] and melt spinning[62-64] can be applied to replace the processes of gas atomization and MA. In combination with controlled oxidation of Y-containing pre-alloys, they may allow the elimination of MA and a significant increase of the throughput to be envisaged, and some examples of their application to ODS alloys are given later.

Additive manufacturing technologies such as selective laser melting (SLM),[65-68] selective laser sintering (SLS)[69] and electron beam melting (EBM)[70] are now well-recognized as capable for efficiently producing complex-shaped, dense parts from metal powders. The use of these techniques to produce parts from mechanically alloyed ODS steel powders provides significant opportunity, as well as additional challenges in preserving the dispersion and size of oxides through the re-melting process. Nonetheless, stability of fine oxide dispersions and encouraging properties when compared with conventional consolidation techniques has been demonstrated.[66-69] These approaches do not replace the MA step, but instead consolidation and forging or machining steps so that the MA powder is converted directly and efficiently (low waste) to an engineering component i.e. making optimum use of the expensive MA powder.

Techniques known from the field of advanced metal-matrix composite fabrication such as three-dimensional fiber deposition (3DFD) have also been applied to achieve an acceptable dispersion of oxide nanoparticles, and to avoid MA.[71]

The hybrid routes for ODS steel fabrication are generally not aimed at producing material that will out-perform conventional PM techniques with respect to the smallest size and most uniform spatial distribution of oxide nanoparticles, or indeed high-temperature properties. The approach is instead to explore the compromises between the relevant properties of the end-product and the suitability for industrial-scale fabrication, including cost issues.

E. *Physico-chemical methods*

Physico-chemical methods can be tentatively classified into ion implantation[72-74], epitaxy including chemical vapor deposition (CVD) and physical vapor deposition (PVD) as well as related variants such as electron beam PVD,[75] molecular beam epitaxy,[76] magnetron sputtering[77] and pulsed laser deposition (PLD). These methods are typically capable of producing samples of lateral dimensions of 10 mm (or less if required) and layers ranging from 10 nm to 1 µm thickness that generally serve as model systems for the investigation of the behavior of interfaces between Fe-

matrix and oxide-particles, e.g. under irradiation. It is important to note that the penetration depth of ion beams is in the correct range to either modify such model systems (e.g. by ion beam mixing) or investigate the role of interfaces on the irradiation behavior.

III.   RESULTS FROM SELECTED RECENT INVESTIGATIONS

A. *Spark plasma sintering*

The work reported here was performed within collaboration between the Helmholtz-Zentrum Dresden Rossendorf (HZDR) and the Fraunhofer Institute IFAM Dresden, Germany. A pre-alloyed ferritic steel powder with the nominal composition Fe-14Cr-1W-0.4Ti-0.3Mn-0.3Si–0.15Ni was produced by Nanoval Germany by means of gas atomization. The powder was mechanically alloyed with 0.6 wt% of Y_2O_3 in a Fritsch Pulverisette P5 planetary ball mill operated under purified argon atmosphere with a ball-to-powder weight ratio of 10:1 and consolidated by means of SPS under vacuum using an FCT-HP D 250/1 spark plasma sintering device from FCT Systeme Germany. Selected elements of the procedure are displayed in Figure 2 along with results obtained by means of SANS. The procedure includes preparation of powder samples for SANS characterization (Figure 2(a)), SPS consolidation of lab-scale samples for process optimization (Figures 2(b) and (c)), SPS consolidation of semi-industrial samples for mechanical characterization (Figure 2(d)), and finally SANS investigations (Figures 2(e) and (f)). Several MA trials were included, some of which are shown in Figure 2(f). The powders after MA were characterized by SEM and SANS[33] (Figure 2(b) and (e)), to achieve a better understanding of the process. Indeed, it was shown by means of SANS that nm-scale oxide particles were already present in the Y_2O_3-free powder, and that as expected the volume fraction of these particles in the as-milled samples increased on Y_2O_3 addition.

MA powders from different sets of MA parameters were consolidated by SPS, using parameters optimized beforehand using criteria such as the avoidance of excessive porosity or any partial melting. The consolidated samples were characterized by means of methods sensitive at the nm-scale, including SANS. The oxide particle radii were between 1 and 10 nm. The particle volume fraction increased as a function of the added Y_2O_3 powder fraction and the intensity of the MA process (Figure 2(f)). It is interesting to note that the total volume fraction of nm-sized particles obtained by SANS for both 30 h/250 rpm (revolutions per minute) and 50 h/250 rpm was greater than the sum of the fraction in the non-strengthened material plus the Y_2O_3 fraction added, indicated as "A+T" in Figure 2(f). This, along with both the measured ratio of magnetic to nuclear SANS (A-ratio) and atom probe tomography results,[78] provides evidence that other elements such as Ti are incorporated into the nanoparticles and that their structure had changed. Indeed, the measured A-

ratios of 2.6 and 2.5 for 30 h/250 rpm and 50 h/250 rpm, respectively, corresponded to calculated A-ratios of $Y_2Ti_2O_7$ ($A = 2.55$) and Y_2TiO_5 ($A = 2.57$), and were significantly different from the calculated A-ratio of Y_2O_3 ($A = 3.26$). Added Y_2O_3 becoming $Y_2Ti_2O_7$ indicates that Y and O were dissociated and dissolved separately instead of surviving as a distinct amorphous phase (compare section II.B). Finally, SPS was successfully scaled up to produce low-porosity compacts of semi-industrial size (Figure 2(d)). Tensile, impact and fracture mechanics testing of miniature samples (Figure 2(d)) is in progress.

B. *Controlled oxidation: description of the method, recent results, future plans*

In conventional ODS manufacturing, MA has the disadvantages of long milling time and contamination (e.g. C, N, excess O) from milling debris and the environment.[60,79-81] The typical consolidation method of the MA powders is HE followed by recrystallization,[14,82] which can cause anisotropic microstructure and disadvantageous directionality in the mechanical properties for some applications.[82] In addition, the MA process is generally considered to be a high cost process resulting in high final material costs.

Rieken *et al.*[79] replaced the MA step by an *in-situ* powder oxidation step during gas atomization, followed by internal oxidation during consolidation and subsequent annealing. The reactive gas atomization involved a Fe-Cr steel melt alloyed with Y and Ti or Hf (all with high oxygen affinity) and atomizing gas containing oxygen. During the process, surface oxidation of the powder particles occured.

The oxidation reactions included the formation of a metastable Cr-enriched oxide shell on the solidified particles. When this powder was then consolidated using HIP, the surface oxidized shell decomposed, released oxygen that diffused through the consolidating compact and preferentially formed Y-rich clusters. By this internal oxidation mechanism, characteristic Y-Ti/Hf -oxide dispersoids formed. The HIPed material was then heat treated to complete the dispersoid formation, and to stabilise the matrix grain microstructure.

The surface oxygen content of the powder particles is a key issue for the subsequent internal oxidation step, with the required oxygen content depending upon the alloy Y concentration. Other challenges are that the gas atomization process is very rapid and the oxidation time is short (typically a few tens or hundreds of milliseconds). In addition, because atomization produces a range of droplet sizes and trajectories, the final oxidation condition may be different for different sized droplets.

An alternative approach to surface oxidized precursor powder is to anneal conventionally inert gas atomized powder alloyed with Y and Ti/Y in a furnace under air. This approach has been

researched and developed at VTT Technical Research Centre of Finland Ltd. The main phases of the process are gas atomizing of Y containing Fe-Cr powder, surface oxidizing of the precursor powder in a furnace, HIP consolidation of the precursor powder and simultaneous internal oxidation. The HIPed material is post-treated by heat treatment, hot working and cold working, as shown in Figure 3.

A complication in this approach is that Y is always lost during the melting and dispatching of the feedstock alloy, even with very high quality vacuum and ceramic systems. Moreover, the remaining Y concentration can be variable. The industry-standard approach is to heavily "over-alloy" with Y in the feedstock alloy. The fluctuation of the Y yield can be mastered over time but adds a significant extra complexity.

Despite these challenges, the surface oxidation of the powder can be well-controlled. For example, a Fe-Cr powder with 8.22% Cr, 1.5% Y and 05 % Ti was oxidized at 753 K (480 °C) in air during a heat-up of 50 minutes, hold of 5 minutes and furnace cooling resulting in an overall oxygen concentration of 0.17 wt.%; a second oxidizing annealing at 773 K (500 °C) involving a heat-up of 50 minutes, hold of 5 minutes and furnace cooling gave an oxygen concentration of 0.28 wt.%.

The consolidation of the oxidized powder is readily achieved by HIP, for instance under a HIP cycle of 1073 K (800 °C)/750 bar/1 h followed by 1423 K (1150 °C)/1000 bar/2h. The step at 1073 K (800 °C) is to enhance the decomposition of the oxide shell of the particles and diffusion of the oxygen. In the resulting as-HIPed microstructure, Y-rich precipitates were dispersed relatively evenly in the microstructure and there were Ti-rich particles in the prior particle boundaries (PPB) (Figure 4). The Ti-rich precipitates consisted of Y, Ti and O, with the smaller ones being mainly binary Y-O based oxides. The high fraction of Ti oxides in the PPBs may have resulted from excessive surface oxidation of the powder, and these Ti-based oxides could not be decomposed in the HIP process.

In the transmission electron microscope (TEM) images in Figures 4(b) and (c), a rather fine sub-structure of ~2 µm could be observed. Oxide precipitates were chained along most of the sub-structure boundaries. Within the sub-structure cells, there was a population of smaller nano-sized particles.

In summary, the manufacture of ODS alloys from Y-containing gas atomized alloys involving powder surface oxidation and then internal oxidation during HIP consolidation has been explored. Controlling the Y concentration during melting is an ongoing technical challenge but nonetheless, controlled post-atomization oxidation of powders under air at 743-773 K (470-500 °C) led to the formation of oxide-rich shells on the powders. Nano-sized oxides were then formed

inside the grains during HIP consolidation. However, most of the oxide precipitates tended to be relatively coarse when compared with PM/MA processed equivalents. Other oxides such as those based on Ti tended to form on the PPBs of the HIPed material. Hot working of the HIPed material helped to break down the PPB structure but the larger oxides persisted. Preliminary mechanical testing is now progressing and will quantify the effects of the coarser oxides on the alloy strength, ductility and toughness.

In a related approach, work in the Department of Materials and Oxford University has investigated if spray forming, closely related to powder atomization, might be useful as a scale-up route to ODS steels. Spray forming is essentially a high cooling rate casting process in which a melt, typically supplied at 25 kg/min is atomized using nitrogen into a relatively coarse droplet spray (mean droplet diameters ~100 μm). Although different size droplets cool and solidify at different rates, on average approximately half of the alloy latent heat is removed from the droplet spray before the trajectories of droplets are interrupted by a rotating substrate, where the droplets deposit, integrate and form incrementally a large billet or preform. Because nucleation of solid has started in the majority of droplets in the flight stage, the remaining billet solidification is highly grain refined and final preform grain sizes are fine-scale (~25 μm even in billet diameters up to 600 mm), grains are polygonal rather than dendritic, and the macro-segregation typical of large diameter castings is almost entirely suppressed. Under optimum conditions, there is usually a residual porosity of ~1% that is closed by TMT. The attraction of spray forming derives from the possible economic and engineering benefits arising from the reduction in processing steps (compared to powder processing methods) and the large billets that can be fabricated.

Figure 5(a) shows a nominal Fe-17Cr-1Y ~15 kg spray formed billet produced at the Department of Materials, University of Oxford. In this preliminary work, there was a small hot tear in the billet centre due to a slight excess liquid fraction at deposition – which can be removed under optimised conditions should the route offer promise for ODS alloys. Inductively coupled plasma optical emission spectroscopy was used to confirm only a 10% Y retention (0.1wt%) in the billet, but as described above, this was not untypical for Y-rich alloy melting and dispensing. Taking a section across the 100 mm billet diameter, room temperature swaging was then used to impart severe cold work to the microstructure via a 50 % reduction. The intention was to induce a high defect density analogous to the MA procedure, while avoiding elevated temperatures and any premature precipitation of Y-rich particles. Millimetre thickness slices of swaged material were then sealed in an evacuated quartz tube together with a sacrificial Fe_2O_3 powder as an oxygen source (similar to Figure 6(b), see later), and heat treated at 800°C for 5 hr. The resulting sub-micron grain structure of the material is shown in the electron back-scattered diffraction (EBSD) image in Figure

5(b). The exceptional refinement despite prolonged thermal exposure was interpreted as indirect evidence of grain boundary stabilisation (Zener pinning), possibly due to the formation of oxides. Subsequent TEM investigations revealed 100-200 nm diameter particles at grain boundaries, as shown in Figure 5(c). Energy dispersive X-ray spectra confirmed the presence of Y in these grain boundaries particles and some residual Y retention within the fine grains (Figure 5(d)). These preliminary data provided sufficient encouragement for further work, which is now focused on spray forming production including modified melting practices for better Y retention, heat treatment optimisation to explore oxide precipitation within grains as well as at grain boundaries, and options for achieving internal oxidation in larger sections.

To explore whether a greater concentration of Y might be retained in a Fe-based meta-stable solid-solution for subsequent internal oxidation, melt spinning of Fe-Cr-Y alloys – which offers cooling rates of up to 10^6 K/s – was also investigated at Oxford University. Pre-alloyed, arc melted Fe-5Y model alloy pellets were induction melted in a BN crucible under an Ar atmosphere (200 mbar) and then ejected using 100 mbar of Ar pressure through a spherical hole (1 mm diameter) in the bottom of crucible and onto a polished spinning Cu wheel rotating at a tangential velocity of 20 m/s. The molten alloy rapidly solidified into 10 to 20 µm thick ribbons, as shown in Figure 6(a). Then, as shown in Figure 6(b), the ribbons were internally oxidized by packing them in a quartz tube with Fe_2O_3 powder, separated by quartz wool, termed a Rhines pack.[83] The quartz tube was pumped to vacuum ($< 1x10^{-4}$ mbar), sealed and then heated to 800°C for 4 hours. The Fe_2O_3 changed colour from red to black, indicating the transfer of oxygen to the ribbons and the corresponding reduction of the Fe_2O_3 to Fe_3O_4. Microstructural analysis in the TEM in Figures 6(c) and (d) shows the formation of a high volume fraction (consistent with high Y starting concentration) of < 50 nm Y-rich particles after internal oxidation.

Based on these encouraging results, investigations are now focused on lower Y concentrations that are more representative of typical ODS steels, the addition of Ti to the starting alloy composition for possible oxide refinement, consolidation of the melt spun ribbons into bulk materials by FAST, and micro-mechanical testing. Although melt spinning may initially appear to suffer from similar restricted scalability issues as PM/MA routes, the large scale processing of Fe-based alloys is already practiced commercially at the multi-tonnes scale for transformer and other specialist alloys.[84]

C. *Contactless alternating-magnetic-field-induced cavitation*

As described earlier, the uniform dispersion of ultrafine oxide particles into the metal melt is one of the crucial steps towards development of an effective LM route to ODS steel fabrication. The

feasibility of a contactless technique of cavitation excitation has been demonstrated recently[59] by way of application of an alternating magnetic field, which, in combination with an axial static magnetic field, gave rise to an alternating body force in a cylinder and, consequently, excitation of cavitation.

For the purpose of demonstration, it is reasonable to primarily apply the method to molten metals exhibiting a low or moderate melting temperature such as Sn, Zn and Al as a first step. For instance, dispersion of Al_2O_3 particles into a Sn melt by means of contactless excitation of cavitation has been demonstrated.[55] More recently, the method was also successfully applied to a stainless steel melt. The onset of cavitation was detected by way of identification of characteristic sub-harmonics in the acoustic signal.[85] This achievement may pave the way towards a contactless method of particle dispersion in molten steels.

IV. SUMMARY AND CONCLUSIONS

The well-established and default process for the production of ODS steels is based on a powder metallurgy (PM) route that depends on an extended mechanical alloying (MA) step in which fine-scale oxides are both intimately mixed with the steel (optimally in the form of pre-alloyed powder) and progressively refined. At extended alloying times (typically 60 hours), discrete crystalline oxides can no longer be convincingly resolved. The property-enhancing precipitation of a high number density but relatively low weight fraction (~0.25wt%) of nano-oxides and/or clusters then occurs during consolidation, heat treatment and any thermo-mechanical processing. In an attempt to avoid the extended and usually costly mechanical alloying step, and to facilitate greater volumes of ODS material, a range of liquid metal and hybrid routes have been developed, including ultrasonic dispersion of particles in the melt, and *in-situ* oxidation approaches based on atomization, spray forming and melt spinning. Although none of the processes have yet reached a maturity where the competitiveness with PM/MA can be assessed, it is concluded there is sufficient encouragement in early results to warrant further work. Critical to these future investigations will be a quantified assessment of the balance between scalability (favoring liquid metal and hybrid routes) and properties (favoring PM/MA).

In terms of consolidation of powders or other finely-divided ODS alloys, along with HE and HIP, SPS and other similar field assisted techniques have now convincingly shown promise for scaling-up to an industrial scale. Although grain size bimodality is reported, the effect of bimodality on bulk properties in real applications, and whether it is therefore a significant area of concern or not, has yet to be clarified. Selective laser sintering and variants also offer significant promise for smaller, niche 3D ODS alloy components.

Given the importance of ODS alloys particularly to the energy generating industry and civil nuclear power in particular, it is suggested that alongside evermore detailed studies on the nano-structure of ODS alloys, and despite the considerable challenges described in this review, efforts to develop alternative processing approaches should be sustained else the understanding and appeal of ODS alloys may remain always constrained to the research laboratory.

ACKNOWLEDGEMENT

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List of Figures

Fig. 1 - Schematic classification of fabrication routes towards ODS steels and model alloys including the standard PM/MA route as reference and several alternative approaches.

Fig. 2 - (a) ODS steel powders in evacuated quartz glass cells (samples 3, 4) for SANS measurements and an empty cell (sample 7) for sample holder correction; (b) lab-scale SPS compact for basic characterization and SPS process optimization; (c) suitable SPS run in terms of pressure p, temperature T and punch displacement; (d) semi-industrial-scale SPS compact and cutting scheme for sample preparation (dimensions in mm); (e) nano-particle size distribution obtained for powders by means of SANS; (f) volume fraction of nano-particles derived from SANS and theoretical fraction if all the nano-particles were cubic yttria.

Fig. 3 - (a) The morphology of the Fe9Cr-powder to be oxidized for ODS. The particle size is: D50 = 33.8 µm and D90 = 76.9 µm. (b) Oxidized powder batch of about 400 g annealed in air at 743 K (470 °C) in the porcelain vessel. (c) Microstructure after HIPing, hot working and annealing. HIP with the parameters of 1073 K (800 °C)/750 bar/1 h followed by 1423 K (1150 °C)/1000 bar/2 h, hot working between 1073 – 1423 K (800 – 1150 °C) 12 times each with about 30 % height reductions and heated four times at 1423 K (1150 °C), and annealing in vacuum at 1123 K (850 °C) for 1 h.

Fig. 4 - (a) SEM-EDS map of the microstructure of the HIPed and internally oxidized sample showing the distribution of Y-rich particles in blue and Ti-rich particles in red. (b) and (c) TEM graphs of the microstructure of the HIPed and internal oxidized sample.

Fig. 5 - (a) 15kg billet of Fe-17Cr-0.1Y produced by spray casting; (b) electron back-scattered diffraction image of the billet microstructure after swaging by 50% at room temperature and internal oxidation; (c) transmission electron microscopy image of Y-rich particles along a grain boundary; and (d) EDX map from the Y Kα1 peak showing Y-rich 100-200nm particles and a weak Y retention in the matrix.

Fig. 6 - (a) Melt spun Fe-5Y ribbons; (b) ribbons before and after internal oxidation using the Rhines pack method; (c) transmission electron microscope (TEM) bright field image of Y-rich particles in internal oxidized ribbons; and (d) Scanning-TEM image of Y-rich particles with chemical analysis from an EDX line scan.