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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 consists of gas atomization of a pre-alloy, mechanical alloying, consolidation and thermal/thermo-mechanical treatment. It was demonstrated in the past that ODS steels with superior property combinations can be produced via the standard route. However, the fabrication process is complex and expensive and the fitness for scaling up to the industrial scale is limited. In the other extreme, small amounts of simple model systems are desirable for special purposes such as modelling-oriented experiments. It is therefore essential to consider a wide and growing range of alternative fabrication routes towards ODS steels and model systems. The paper is aimed at reviewing the state of the art and identifying promising new fabrication routes towards ODS steels depending on the particular purpose of the material. The standard PM route for the fabrication of ODS steels is included as a reference. Hot isostatic pressing, hot extrusion and pulsed-current-assisted sintering are frequently applied consolidation techniques. Hybrid routes such as melt spinning/consolidation are promising approaches to scale the fabrication up towards larger volumes and higher throughput of fabricated material. Although a uniform distribution of nm-sized oxide particles cannot currently be achieved in the melt, new innovation on the liquid metal (LM) route towards nanoparticle-strengthened alloys is included in the review. Important aspects are the pre-processing of powders and post-processing of compacts including thermomechanical treatment and severe plastic deformation. Finally, physico-chemical methods such as ion beam synthesis offer interesting perspectives for the fabrication of model systems. Selected examples of recent progress are highlighted.

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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 are considered to improve creep strength, thus 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 nuclear environments. It is interesting to note that different fabrication routes such as mechanical alloying (MA) of steel/oxide powder blends[1] or controlled oxidation of Y-alloyed steel powders were considered from the very beginning,[9] see also.[2] Indeed, the notion of MA did not come up until 1970.[10]

After commercial ODS products had become temporarily available in the 1990s, two lines of scientific effort received special interest. On the one hand, composition and processing parameters were varied to obtain optimum microstructures and/or optimum mechanical properties. For instance, it was shown that the addition of about 0.4 wt% Ti to the master alloy gives rise to a reduction of the oxide-particle size from about 20 nm to about 2 nm[5] and thereby a significant improvement of creep resistance. Consolidation techniques included hot-isostatic pressing (HIP), hot extrusion (HE) and, more recently, pulsed-current-assisted sintering also known as spark plasma sintering (SPS). The role of post-processing of compacted products including thermo-mechanical treatment was also investigated. On the other hand, much work was motivated by the desire to simplify the overall process, to make it less expensive, and to allow scaling up towards industrial scales to be achieved. Clearly, scaling up of the process and optimization of the properties have to end up in certain compromises.

In the last few years the number of approaches applied to fabricate ODS steels or dispersion-hardened model alloys has been increasing and diversifying rapidly. A comprehensive source of information 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 the 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 first objective of the present study is to summarize and pigeonhole reported approaches of ODS steel fabrication in order to simplify orientation in the field. In doing so, broadness of the review is preferred over in-depth evaluation of future perspectives. Four major groups of approaches are distinguished. These are the powder metallurgy (PM) route, the liquid metal (LM) route, hybrid routes based on elements from both the PM and LM route, and physico-chemical methods, the latter focused on the fabrication of model alloys.

The second objective of the paper is to highlight few selected results recently obtained within the consortium of the pilot project AFROS introduced above. Pulsed-current-assisted sintering (SPS), which is capable of reducing sintering time and limiting coarsening of the microstructure, controlled oxidation of Y-alloyed gas atomized powder as an approach to avoid the MA step, and contactless alternating-magnetic-field-induced cavitation as a milestone on the LM route are addressed.

I. FABRICATION ROUTES TOWARDS ODS STEELS AND MODEL ALLOYS

A. *The standard PM route*

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

- selection of the steel composition or its variation,
- gas atomization of the steel to be dispersion hardened (master alloy),
- blending of the steel powder and suitable oxide powders, e.g. commercial Y_2O_3,
- mechanical alloying (MA) 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).

Over the years continuing progress has been achieved on each of these steps. Some of them will be highlighted in the next section. For the purpose of the identification of promising alternative fabrication routes, the standard PM 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 MA and HIP.

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

B. *Recent progress related to the PM route*

From the viewpoint of the composition of ODS steels, recent efforts were 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 about 0.4 wt% Ti turned out to be crucial in both cases to achieve ultrafine particle dispersion.[5] Cladding tubes as one of the most advanced potential nuclear energy applications were successfully produced from both variants.[14] Comprehensive microstructural and mechanical characterization of the tube materials is in progress within a running European Project. The effect of several types of oxide powders on the microstructure and mechanical properties of HIPed Fe-13%Cr steel was considered on the lab scale.[15]

Recent progress in gas atomization of liquid metals was achieved using a De 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 type of mill (attritor mill, planetary ball mill), milling atmospheres (argon, hydrogen) and milling parameters (revolutions per minute, ball to powder ratio, milling time) were varied individually or in combination. These influence factors are typically cross-correlated with one another and with other parameters such as steel composition and initial powder quality. Moreover, they may depend on the details of the milling equipment used. In other words, *the universal* set of parameters that guarantees optimum properties does not exist. It was shown that during a typical milling run the yttria powder particles do not dissolve but become ultrafine and defective so that they appear to be X-ray amorphous.[18] This seems to be in contrast to the earlier view telling that Y and O completely dissolve in the Fe alloy matrix.[6] However, a closer look at the latter results shows that it is refinement to below the detection limit of incoherent SANS, what was observed. This would be consistent with the former conclusion.

The consolidation techniques of HIP and HE have been well established for a long time and are not considered below. More recently, SPS and other field-assisted consolidation techniques have received increasing interest. SPS was applied to FeAl intermetallics,[19] iron,[20] Fe-9%Cr,[21-23] Fe-14%Cr-X,[24-28] and Fe-16%Cr-3%Al.[29] SPS was shown to be suitable for the fabrication of semi-industrial batches of ODS steel (~0.5 kg).[30,31] A typical (but not limited to SPS) observation are bimodal grain size distributions.[20,28,32,33] The abnormal grain growth experienced by the larger-size fraction was rationalized by several authors.[32,34] It is interesting to note that bimodal grain size distributions were suggested to be a potential basis for favorable property combinations of nanostructured materials in general[35,36] and for ODS steels in particular.[37]

Closely related methods of field-assisted sintering have also been applied to the consolidation of ODS steel powders. Field-assisted hot pressing (FAHP), using a low-frequency alternating current, was applied successfully by Garcia-Junceda et al..[38] Effectiveness of microwave sintering over conventional vacuum sintering was reported by Yao et al.[39] for a 14%Cr ODS steel.

The application of friction consolidation to process ODS steels was suggested by Catalini et al.[40,41] to tackle the downsides of the conventional processing route. These authors demonstrated the ability of friction consolidation to achieve ultrafine oxide precipitation at potentially reduced processing costs.

The role of different kinds of TMT was investigated by Oksiuta et al..[42] Among HE, hot pressing and hot rolling of the as-HIPed ODS steel, the rankings with respect to texture, strength and fracture seem to be inconsistent indicating that more work is needed to find the optimum TMT parameters.

Finally, severe plastic deformation (SPD) processes such as equal channel angular extrusion (ECAE) or pressing (ECAP) and high-speed hot extrusion (HSHE) were applied to ODS steels.[12,43,44] Both HSHE and ECAE of the as-HIPed material were shown to give rise to a significant microstructure refinement and extra hardening.[43,44] However, SPD applied to PM ODS steels seems to be counterproductive from an economic point of view, because the expensive PM route itself is already aimed at producing a nanostructured alloy and contains elements of SPD in the MA process .

C. *The LM route*

Track of melt processing of ODS steels has never been lost over the decades.[45] A popular students' examination question related to ODS steels is, why the oxide powder is not added directly to the metallic melt. Partial answers may refer to limited wettability of the oxide particles, the tendency to agglomerate already before the introduction of the particles into the melt or at the surface of the melt, and the limited effectiveness of mixing due to different gravimetric properties. However, the attractiveness of the LM route in terms industrial-scale production as compared to the PM route makes people stay thinking about possible LM-based approaches.[45,46]

Because of the challenges mentioned above and others, it is natural that work in progress concentrates on the laboratory scale and basic aspects. As the oxide-particles, once introduced into the melt, cannot be refined anymore, nanoscale oxide-powders have to be prepared, whereas μm-scale oxide powders may be sufficient for mechanical alloying. In this respect, nano-milling was shown to produce finer oxide powders than conventional planetary ball milling.[47]

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 was suggested and investigated.[48] Wettability of oxide particles in the metallic melt is another pre-requisite in order to allow bonding between the nanoparticles and the metal matrix to be achieved. A systematic study of the contact angle of iron-chromium droplets onto yttria substrates was reported.[48] The generally weak wettability of yttria particles observed for the Fe-Cr system was shown to be improved by the addition of metallic yttrium into the melt.[49] Finally, the quality of dispersion in a rapidly solidified cast iron-yttria composite was assessed.[49]

Any casting technology for nano-composites or ODS steels requires the solution of several complex problems. The particles need to be brought into the bulk of the metal melt, they need to be finely dispersed and they have to stay intact during solidification. For the particle insertion several approaches exist such as filling into hollows made in the initial metal sample, full or partial mechanical alloying, electrochemical particle co-deposition as well as intense stirring through the surface of the molten metal. The latter can preferably be done in a contactless way by magnetic stirring[50] 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.[51]

Once nanoparticles are incorporated into a metal melt, strong capillary and inter-particle forces tend to agglomerate them to, at least, micro-sized clusters. In order to avoid this, a powerful agitation of the melt is needed. For light metals ultrasound is typically used for that purpose.[52,53] The mechanism behind is related to cavitation. Collapsing cavitation bubbles produce powerful shocks and microjets which are capable to overcome the forces holding together clusters of nano-particles. In other words: just a fluid-dynamic stirring of the melt will yield, at best, a fine dispersion of micro-particles, but not of nano-particles. The contactless magnetic excitation of acoustic cavitation in liquid metals based on a superposition of a steady and an alternating magnetic field has recently been demonstrated.[54]

After the particles are successfully inserted and dispersed, the sample has to be solidified. Several problems may arise. If the particle life-time under conditions of the molten metal is too short, they may react with the matrix elements or dissolve. If, in turn, the particle affinity to the metal is insufficient, then they may be pushed ahead of the advancing solidification front or they may re-agglomerate upon collisions. As mentioned above, the wettability of the particles plays a decisive role hereby.

The application of those ideas to ODS high-Cr steels, scaling down to nm-sized particles, and scaling up to industrial cast volumes is expected to pose future challenges.

D. *Hybrid routes*

The notion of hybrid routes refers to the idea that fabrication methods of ODS steels may contain elements from the PM and the LM route. However, it can be understood in a broader context to contain elements of chemically, physically or metallurgically different principles.

As already mentioned in the introduction, the idea of controlled oxidation of a Y-alloyed 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 save the MA step of the standard PM route.[55] As a remarkable example, the high-throughput process of gas atomization reaction synthesis (GARS) of Fe-16%Cr alloy powders was reported by Rieken et al..[56] Internal solid-state O exchange reactions between metastable Cr-rich oxides and Y-enriched intermetallic precipitates give rise to a uniform distribution of nm-sized mixed oxides.

Methods such as spray forming[57] and melt spinning[58,59] can be applied to replace the processes of gas atomization and MA.[60] 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. Clearly, these are important steps towards an industrial-scale fabrication.

Additive manufacturing technologies such as selective laser melting (SLM),[61-63] selective laser sintering (SLS)[64] and electron beam melting (EBM)[65] are well recognized to be capable of efficiently producing complex dense parts from metal powders. The use of these techniques to produce parts from mechanically alloyed ODS steel powders poses additional challenges. In some cases the material was demonstrated to maintain fine oxide dispersions and encouraging properties as compared to conventional consolidation techniques.[61,62,64] Instead of replacing the step of MA, these methods are rather aimed at utilizing the advantages of additive manufacturing and rapid prototyping, i.e. to make optimum use of the expensive MA powder.

Techniques known from the advanced field of metal-matrix composite fabrication such as three-dimensional fiber deposition (3DFD) were also applied to achieve a dispersion of oxide nanoparticles and avoid MA.[66]

The above mentioned hybrid routes of ODS steel fabrication are not aimed at outperforming conventional PM techniques with respect to smallest size and uniform spatial distribution of oxide nanoparticles or high-temperature properties. The challenge is rather to find good compromises between the relevant properties of the end-product and suitability for industrial-scale fabrication including cost issues. Such a compromise clearly depends on the considered application.

E. *Physico-chemical methods*

Physico-chemical methods can be tentatively classified into ion implantation[67-69] and epitaxy including chemical vapor deposition (CVD) and physical vapor deposition (PVD) as well as related variants such as electron beam PVD,[70] molecular beam epitaxy,[71] magnetron sputtering[72] and pulsed laser deposition (PLD). These methods are typically capable of producing samples of small size or layers of small thickness, which may serve as model systems for the investigation of the behavior of interfaces between Fe-matrix and oxide-particles, e.g. under irradiation, and they were applied in that sense in the above-mentioned publications.

II. RESULTS FROM SELECTED RECENT INVESTIGATIONS

A. *Spark plasma sintering*

A prealloyed 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. It 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 small-angle neutron scattering (SANS). The procedure includes preparation of powder samples for SANS characterization, Figure 2(a), SPS consolidation of lab-scale samples, Figures 2(b) and (c), for process optimization, SPS consolidation of semi-industrial samples, Figure 2(d), for mechanical characterization, and SANS results, Figures 2(e) and (f). Several MA trials were included as partly indicated in Figure 2(f). The MA powders were characterized by SEM and SANS, Figures 2(a) and (e), to achieve a better understanding of the process. Indeed, it was shown that nm-scale oxide particles are already present in the Y_2O_3-free powder, but that the volume fraction of these particles in the as-milled samples increases due to Y_2O_3 addition. However, this evidence was insufficient to predict the quality of the consolidated material.

Therefore, powders exposed to milling using different sets of MA parameters were consolidated. The SPS parameters were optimized beforehand using criteria such as to avoid excessive porosity or partial melting. The consolidated samples were then characterized by means of methods sensitive at the nm-scale including SANS. It was shown that the particle radii are between 1 and 10 nm. The particle volume fraction increases as a function of added Y_2O_3 powder fraction and 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 is higher than the sum of the fraction in the non-strengthened material and the theoretical Y_2O_3 fraction indicated as A+T in Figure 2(f). This, along with the A-ratio of SANS and atom probe tomography results discussed elsewhere, gives evidence that other elements such as Ti are incorporated in the nanoparticles and that the structure has changed. Finally, the procedure turned out to be successful the fabrication of low-porosity compacts of semi-industrial size. Tensile, impact and fracture mechanics testing of sub-sized 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 the long milling time and contamination (e.g. C, N, excess O) from milling debris and gas environment.[54,73-75] The typical consolidation method of the MA powders is HE followed by recrystallization, which can cause anisotropic microstructure and disadvantageous directionality in the mechanical properties. In addition, the MA process has high costs resulting in extremely high material costs.

Rieken et al.[73] reported a manufacturing method where MA is substituted by powder oxidation during gas atomisation and internal oxidation during consolidation and post-annealing. In this method the ODS precursor powder is produced by reactive gas atomisation of Fe-Cr steel melt alloyed with elements of yttrium and titanium or hafnium which all have extremely high oxygen affinity. In atomizing by oxygen containing gas, surface oxidation of the powder particles occurs.

During atomisation chromium builds up a metastable Cr-enriched oxide shell on the solidified particles. When the surface oxidized powder is consolidated using HIP, there is time for the oxide shell to decompose, released oxygen to diffuse towards the particles' centre, and yttrium enriched clusters to oxidise. By this internal oxidation mechanism Y-Ti/Hf -oxide dispersoids form. The HIPed material can be post-heat treated to complete the dispersoid formation and control the matrix microstructure. Thermo-mechanical treatments are used to enhance the material properties.

The surface oxygen content of the powder particles is a key issue in the internal oxidation. The desired oxygen content depends on the yttrium content. Controlled oxidation during gas atomisation may have challenges, as the gas atomisation process is very rapid and the oxidation time remains short. In addition, the melt droplets and subsequent solidified powder particles probably tend to oxidise differentially, because the oxidisation rate is also dependent on the location in the spray and small particles cool down more quickly. A different approach to surface oxidized precursor powder is to anneal the conventionally inert gas atomized powder alloyed with yttrium and titanium/hafnium in the furnace under air atmosphere. This approach has been researched and developed at VTT. The main phases of the process are gas atomising of yttrium containing Fe-Cr powder, surface oxidising 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. In Figure 3 there are snapshots of the process.

The controlled alloying of the gas atomized powder with yttrium has proved out to be difficult. In the melting and alloying of the batch for the atomisation, the yield of the yttrium is low and fluctuating. Anyhow, the melt batch must be heavily over-alloyed with yttrium. The fluctuation of the yield should be able to master by tight process practise but is still an unsolved issue.

The surface oxidation of the powder can well be controlled in air convection furnace. For example, Fe9Cr-powder with 8.22 % Cr, 1.5 % Y and 0.5 % Ti was oxidized at 753 K (480 °C) in air atmosphere during heat-up of 50 minutes, hold of 5 minutes and furnace cooling resulting to oxygen content of 0.17 wt.%. After the second oxidising annealing at 773 K (500 °C) during heat-up of 50 minutes, hold of 5 minutes and furnace cooling, the oxygen content was 0.28 wt.%.

The consolidation of the precursor powder and internal oxidation can well be done in HIP. For instance, the HIP program of 1073 K (800 °C)/750 bar/1 h followed by 1423 K (1150 °C)/1000 bar/2h is valid. The program includes a step at 1073 K (800 °C) to enhance the decomposition of the oxide shell of the particles and diffusion of the oxygen.

In the as HIPed microstructure there can be seen Y-rich precipitations dispersed quite evenly in the microstructure and Ti-rich in the prior particle boundaries (PPB), see Figure 4. The Ti-rich precipitates consist of Y, Ti and O, the smaller ones being mainly pure yttrium oxides. The high amount of Ti oxides in the PPBs may result from the too heavy surface oxidation of the powder. The formed Ti oxides cannot decompose in the HIP process.

In TEM micrographs (Figure 4), rather fine sub-structure of ~2 µm can be observed. Oxide precipitates are chained along most of the sub-structure boundaries. Within the sub-structure cells there are a number of nano-size particles.

As conclusion of the first trials and for the future prospects it can be stated that:

- ODS can be manufactured from yttrium alloyed gas atomized powder without mechanical alloying using separate powder oxidation and internal oxidation during HIP consolidation
- In the melting and alloying of the atomized melt, Y tend to have low and fluctuating yield, probably because of the reactions with the alumina crucible, although it is yttria coated.
- The alloying and melting practice has to be developed further (protection of the melting crucible, use of master alloys in melt charge, optimization of the over alloying, fast melting and casting).

- Well controlled powder oxidation can be performed in a common air convection furnace at 743 – 773 K (470 – 500 °C).
- Nano-size oxide dispersions will form inside the grains during the HIP consolidation of the pre-oxidized powder. However, most of the oxide precipitations tend to be relative coarse compared to those typically in ODS steels presented in the literature.
- Number of coarse micron-size oxides and oxide clusters tend to form in the PPBs of the HIPed material.
- Hot working of the HIPed material can broke the PPBs and may be used to homogenize the oxide distribution but probably not to reduce the size of the oxides.
- Alloying in gas atomisation and surface oxidation has to be optimized to have optimum and more controlled content of Y, Ti/Hf and O for finer oxide dispersoids.
- Basic mechanical testing shall be carried out to evaluate the strength, ductility and toughness, and the final quality of the ODS material.

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

Uniform dispersion of ultrafine oxide particles into the metal melt is one of the crucial steps towards an LM route of ODS steel fabrication. It is well known that ultrasonic cavitation represents an encouraging option to reach this partial goal. However, cavitation also degrades the vibrating surface transmitting the ultrasound into the liquid, particularly for high-temperature metal melts. A contactless technique for cavitation excitation is therefore desirable.[54] The feasibility of the latter has been demonstrated recently[54] by way of application of an alternating magnetic field, which, in combination with an axial static magnetic field, gives 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 tin, zinc and aluminium. For instance, dispersion of Al_2O_3 particles into a tin melt by means of contactless excitation of cavitation was shown to work.[51] More recently, the method was also successfully applied to the situation of a stainless steel melt. The onset of cavitation was detected by way of identification of characteristic sub-harmonics in the acoustic signal.[76] This achievement may pave the way towards a contactless method of particle dispersion in molten steels. However, the LM route of ODS steel fabrication remains a challenge.

SUMMARY AND CONCLUSION

Reported approaches of ODS steel fabrication were summarized and tentatively pigeonholed. Emphasis was put on a range of alternative fabrication routes independently of their present status of advancement. Current activities in the field and obtained results were highlighted. Along with HE and HIP, SPS represents a third consolidation route fit for scaling up to an industrial level. Improved control of the resulting pronounced bimodality of the grain size distribution has to be envisaged. Basic problems are being solved on the liquid metal route in order to achieve suitability for ODS steel fabrication. A variety of hybrid routes indicate potential for achieving a tradeoff between economy of the fabrication process and quality of the end product.

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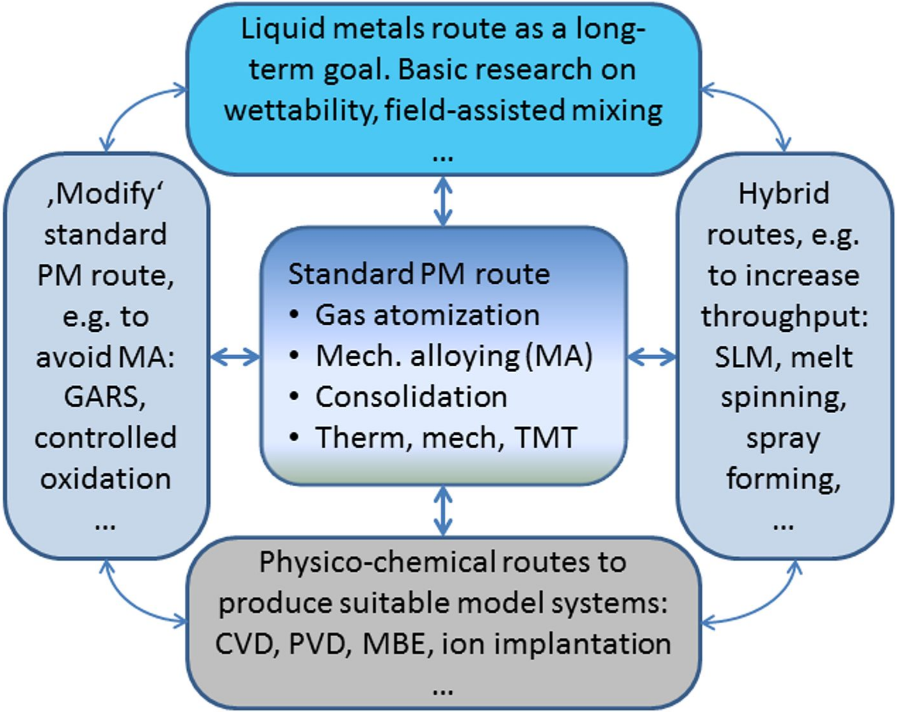
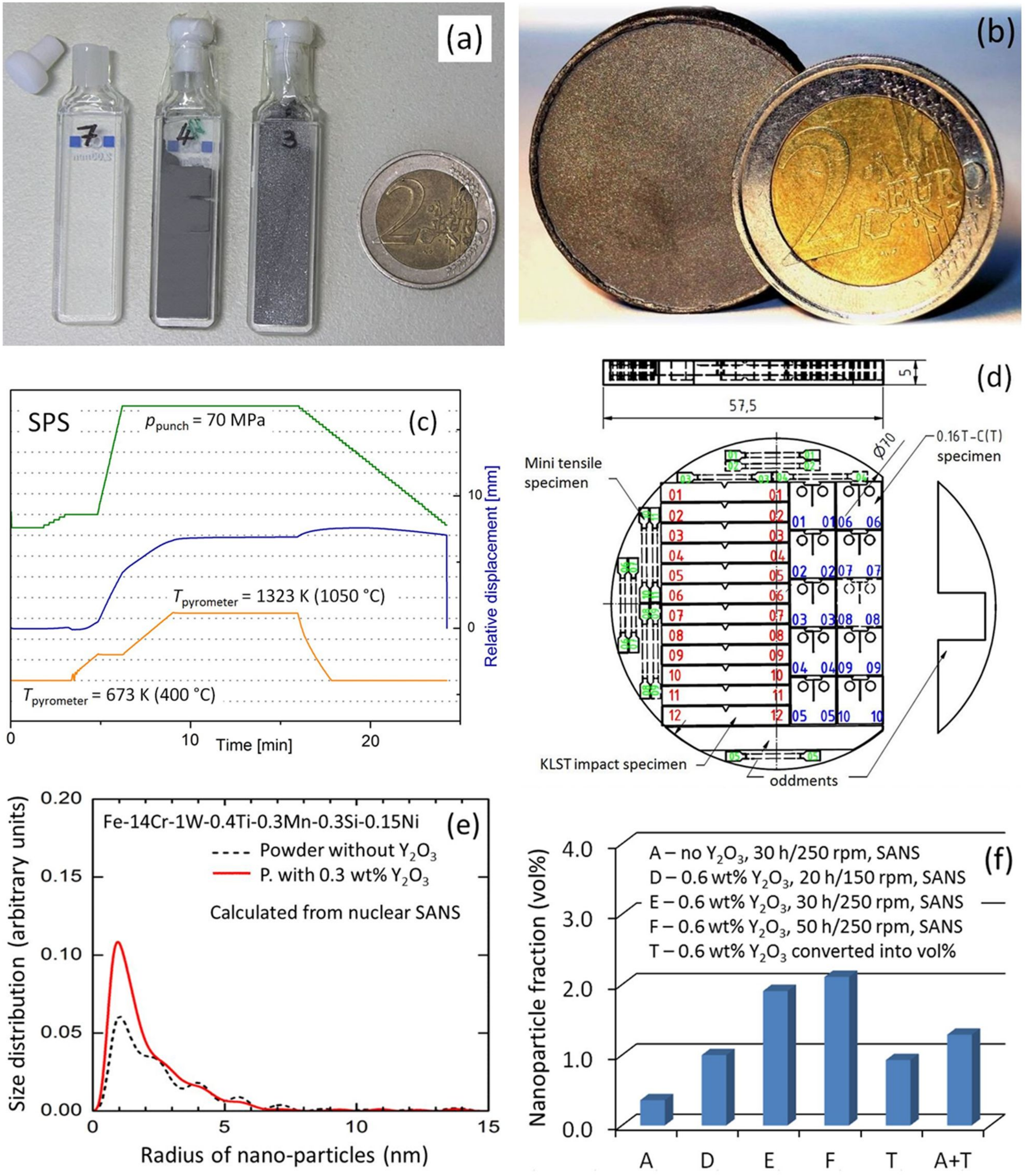
List of Figures

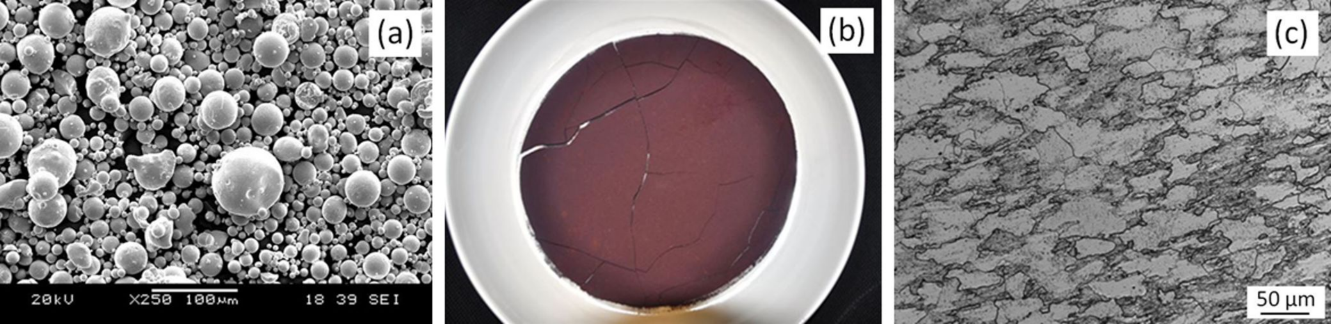


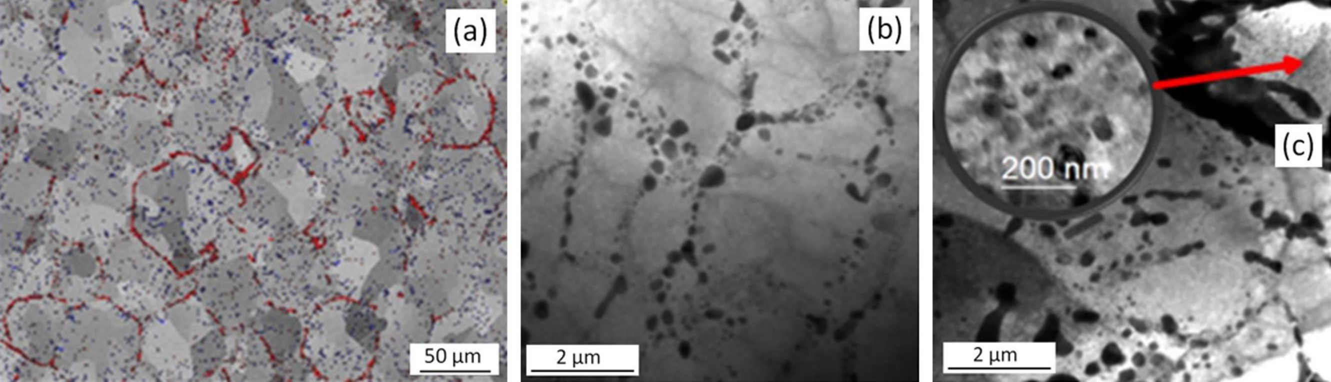
Abbreviations:

PM – Powder metallurgy
MA – Mechanical alloying
TMT – Thermomechanical treatment
GARS – Gas atomization reaction synthesis
SLM – Selective laser melting
CVD – Chemical vapor deposition
PVD – Physical vapor deposition
MBE – Molecular beam epitaxy



(a)
7
4
3
(b)
SPS
p_{punch} = 70 MPa
(c)
$T_{\mathrm{pyrometer}}$ = 1323 K (1050 °C)
$T_{\mathrm{pyrometer}}$ = 673 K (400 °C)
Relative displacement [mm]
Time [min]
(d)
57,5
5
Ø70
0.16T-C(T) specimen
Mini tensile specimen
KLST impact specimen
oddments
Fe-14Cr-1W-0.4Ti-0.3Mn-0.3Si-0.15Ni
(e)
Powder without Y_2O_3
P. with 0.3 wt% Y_2O_3
Calculated from nuclear SANS
Size distribution (arbitrary units)
Radius of nano-particles (nm)
(f)
A – no Y_2O_3, 30 h/250 rpm, SANS
D – 0.6 wt% Y_2O_3, 20 h/150 rpm, SANS
E – 0.6 wt% Y_2O_3, 30 h/250 rpm, SANS
F – 0.6 wt% Y_2O_3, 50 h/250 rpm, SANS
T – 0.6 wt% Y_2O_3 converted into vol%
Nanoparticle fraction (vol%)
A
D
E
F
T
A+T


(a)
20kV
X250 100μm
18 39 SEI
(b)
(c)
50 μm


(a)
50 µm
(b)
2 µm
(c)
200 nm
2 µm