

CONSOLIDATION OF NANO-OXIDE DISPERSION STRENGTHENED AUSTENITIC STAINLESS STEELS FOR HIGH TEMPERATURE APPLICATIONS – A REVIEW

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Abstract: *Austenitic stainless steels play vital role in high temperature applications. The components used in fission reactors should have better tensile properties at high temperature. To overcome the issues raised by conventional steels, a new group of steels known as oxide dispersion strengthened (ODS) steels are created. Nowadays, two groups of ODS steels such as the ferritic and austenitic ODS steels are being used. The present work mainly focusses on the processing of ODS austenitic steels. The consolidation processes adopted for ODS austenitic steels are discussed in detail. The microstructural features and precipitation of nano-oxides during compaction are also reported. In addition to this, the mechanical properties such as tensile strength and elongation values obtained by various manufacturing processes are also discussed.*

Key words: *Oxide Dispersion Strengthened, Austenitic Steel, Consolidation, Nano-oxides, Tensile Strength and Elongation.*

1. INTRODUCTION

Behavior of metals at high temperature is of essential importance to many industries such as aerospace, petrochemical and nuclear power plants. The ability to operate at elevated temperature is a decisive factor for the materials used as a structural component in these industries. Swelling resistance and high operating temperature are the common objectives of nuclear reactors, calling for advanced high strength materials with superior corrosion resistance [1]. The stainless steels that are used in high temperature applications are as follows.

1.1 Ferritic/Martensitic Stainless Steels

Ferritic and martensitic steels were first developed (in 1970s) for high temperature applications for nuclear and petrochemical industries due to their higher chromium content (10-12 Wt. %) caused by superior thermal properties. These steels are well balanced materials with better functional properties and field

experience [2]. Even though the ferritic/martensitic grades have been used in the power generation industry when relating them to nuclear (GEN IV) plants, many additional concerns are necessary. The important point is operation temperature. The operating temperature range of nuclear power plants will be 500 °C to 550 °C. This difference causes significant changes in the properties of ferritic and martensitic steels. The property such as creep strength is the most important while operating temperature is above 500 °C. In addition to this, the structural materials will experience many of the challenges as follows: (1) high corrosive environments, (2) high operating temperatures and (3) neutron radiation [3].

The materials used in high temperature applications require some of the characteristics such as better high temperature mechanical properties, irradiation resistance, and close dimensional tolerance against the void swelling. Ferritic and martensitic steels are structured by a balanced microstructure of ferrite stabilizing

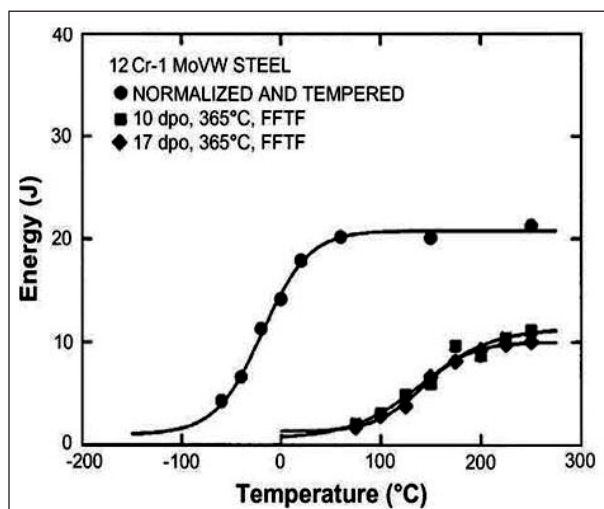


Fig 1. Impact Energy vs Temperature for F/M steels [2]

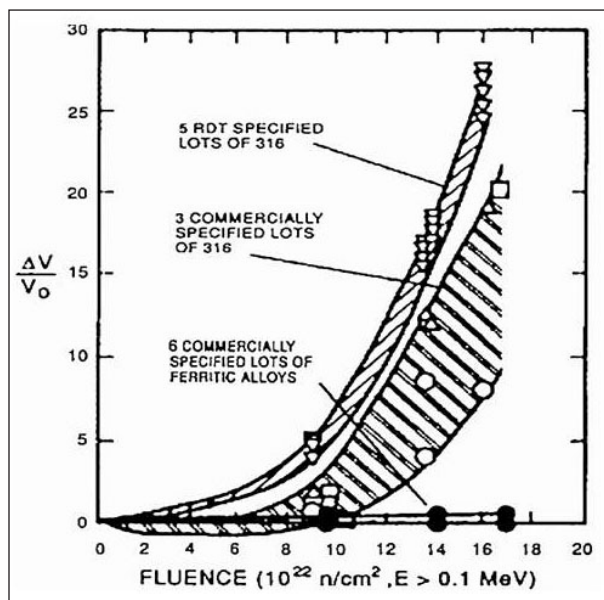


Fig 2. Void Swelling Resistance in Austenitic and Ferritic Steels [2]

elements and austenite stabilizing elements. However, when the temperature is above 700 °C, then due to transformation between ferrite and martensite, a tempered martensite structure takes place. These steels are generally used in nuclear reactors previously due to excellent void swelling resistance. On the other hand, there are some worries concerning poor long term creep resistance and embrittlement as seen in Fig.1. Therefore, researchers are moving towards austenitic stainless steels as alternate materials for high temperature applications.

1.2 Austenitic Stainless Steels

Austenitic stainless steels have attracted more

attention during the last decade due to their unique properties. Austenitic grades have excellent resistance to corrosion and oxidation coupled with good creep resistance. Austenitic stainless steels have been widely used in biomedical, architectural and automotive applications due to excellent fabrication and toughness properties. However, void swelling remains a main performance issue as shown in Fig.2.

The irradiation and void swelling may have important implications on the dimensional stability and performance of the nuclear reactor which limits the usage of conventional steels. Researchers are identifying the solution for the issues raised by conventional stainless steels. It is stated that the oxide dispersion strengthened alloys are promising materials for high operating temperature in nuclear power plants [2]. The nano-sized dispersion of oxide materials in the steel matrix causes drastic improvements in the properties of conventional steels. Reports relating to austenitic steels suggesting that the high density dislocations lead to better void formation resistance. The introduction of nano-sized Y_2O_3 to the austenitic steel matrix by dispersion strengthening mechanism is considered to be an effective way of improving the high temperature properties. Oxide dispersion strengthened (ODS) materials have been shown good high temperature properties and irradiation resistance [4]. The yttria particles in the austenite matrix will hinder grain growth, grain boundary sliding and re-crystallization thereby improving the irradiation resistance and high temperature strength.

The first ODS steel developed in 1960 and has been continuing since. A traditional casting process has a limitation in particle agglomeration caused by the addition of nano sized ODS materials. Metallurgical scientists overcome this drawback by the adoption of powder metallurgy (P/M) processes. Generally oxide dispersion strengthened austenitic stainless steels are manufactured by powder metallurgy route, including mechanical alloying and compaction of powder blends.

A lot of research has been carried out on ODS ferritic steels but there is limited knowledge base on ODS austenitic steels. After 1990s, ODS austenitic steels becomes commercial and one scientific effort received special interest. That is processing methods for ODS austenitic stainless steels. In this paper, various consolidation (manufacturing) methods for oxide dispersion

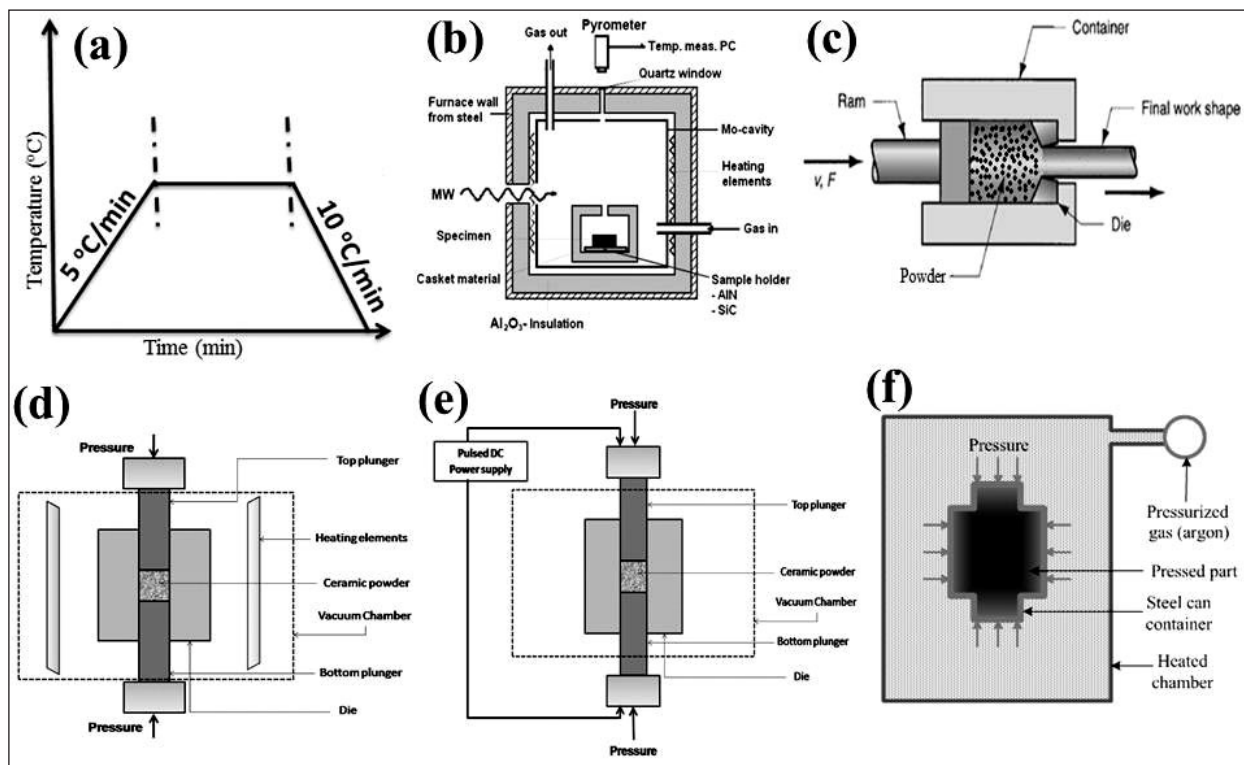


Fig 3. Manufacturing Processes: (a) CS; (b) MS [5]; (c) EXTR [6]; (d) HP [7]; (e) SPS [7]; (f) HIP [8];

Table 1: Sintering Parameters for CS Process

Sintering parameters	Optimum values
Compaction pressure	550 to 650 MPa
Sintering temperature	1100 to 1250 °C
Sintering time	60 to 120 min
Cooling rate	5 to 10 °C/min
Sintering atmosphere	Vacuum

strengthened austenitic steels have been reviewed. The microstructure and mechanical properties of ODS austenitic stainless steels as obtained by various consolidation processes also presented.

2. MANUFACTURING METHODS

ODS austenitic steel powders are normally synthesized by the mechanical alloying (MA) process. MA was developed initially to produce high strength and lightweight materials in the aerospace industry. It is a powder blending process involving repeated fracturing and re-welding for processing equilibrium and non-equilibrium alloy phases through a high energy ball mill [4]. The milled powders of ODS composition are compacted by different consolidation processes.

2.1. Conventional Sintering (CS)

Conventional sintering (CS) generally consists of two steps. The first step is powder compaction. The powder compaction can be done at room temperature in a cylindrical die using a universal testing machine. The second step is sintering process. Sintering process can be carried out in a muffle furnace of controlled atmosphere. Compaction pressure, sintering temperature and rate of cooling are the three essential parameters for effective implementation of conventional sintering process [9].

Jain [10] studied the conventional sintering process during the compaction of yttria (Y_2O_3) added austenitic stainless steel. The CS processed samples displayed better sintered density and hardness. The highest sintered density obtained by CS was 87 to 89 % of theoretical density. Moreover, the oxide dispersion strengthened austenitic steel powders are compacted by this process [11]. The uniform dispersion of the yttria particles in the microstructure of austenitic steels was achieved by this process. The optimum values of sintering parameters reported by researchers for compacting the ODS austenitic steels are shown in Table 1. However, the lower sintering rate and higher sintering time (Fig.3a) are the major drawbacks in this process

2.2. Microwave Sintering (MS)

In order to overcome the drawbacks of conventional process, material scientists identified the process of microwave sintering (MS). Microwave sintering is different from conventional sintering, which involves absorb the electromagnetic energy volumetrically, and transform into heat. It is a process of radiant heating followed by transfer of thermal energy to the inside of the sample being placed in the microwave furnace Fig.3b). In MS, the electromagnetic energy can be converted into thermal energy which is highly efficient. In CS, the surface of the material is heated first, followed by entire surface, whereas heat is generated in the inner surface of the material in the case of MS.

Annamalai, et al., [12] analyzed the microwave sintering process during the compaction of yttria (Y_2O_3) added austenitic stainless steel. He reported that the MS sintered samples exhibited better densification during microwave sintering when compared to conventional sintering. However, low sintering temperature and short sintering time are the main drawbacks which significantly decrease the hardness of austenitic steels compared to conventional sintering.

2.3. Extrusion (EXTR)

It is a process of forcing the powder blends under a high temperature and pressure. The powders after milling were filled with cans, de-gassed at the 500 °C for 4 hours and then consolidated by extrusion process [13]. The cans were heated up to 6 hours to ensure uniform temperature before extrusion. The extrusion process is a one step process with an operating temperature of 1100 °C as reported in [13]. The process of extrusion is shown in Fig. 3c. High equipment setup and need of secondary processes are the major drawbacks in the extrusion process.

2.4. Hot Pressing (HP)

This process is initially developed for fabricating the hard and brittle materials. Later, it is used for compacting the ODS steels due to attractive properties achieved by this process. The pre-alloyed and elemental powders filled in the graphite die that withstand resistance heating up to a temperature around 2300 °C and a pressure of 60 MPa pressure. Hot pressing (HP) addresses many of the issues by conventional process such as long sintering cycle and porosity of samples. HP is a high pressure P/M process for consolidating the

ODS steel powders at high temperature to induce high creep strength in the material. The schematic representation of HP process is shown in Fig.3d. In HP process, the pressure and temperature simultaneously applied to compact the powder blends. The pressure is applied, hydraulically and the heating is generated using graphite resister under vacuum atmosphere.

Researchers pointed that vacuum hot pressing is an excellent method for consolidating the ODS austenitic stainless steel powders. Sornin, et al., [14] studied the fabrication of ODS steels by hot uni-axial pressing. He reported the powders were fully compacted and homogeneous microstructure which does not present any gradients of hardness. One of the issues is crystallographic mis-orientations caused due to plastic strain generated during the rapid compaction.

2.5. Spark Plasma Sintering (SPS)

Spark plasma sintering (SPS) is also known as pulse electric current sintering. It is a novel technique by subjecting the green compact to arc discharge generated by a pulse direct current. This new technique sinters a material powder on a microscopic level and accelerates material diffusion. It is the most effective densification process for compacting nano-structured materials. Researchers proved that the SPS sintered samples achieved full densification and small grain size, which substantially improves functional properties of materials [7].

The experimental setup of SPS process is shown in Fig.3e. The SPS process suppresses the grain growth and fully dense components by rapid heating at high temperature. In this process the temperature measured in the inside sample is more when compared to the temperature on the die surface. During the process of SPS the oxide contamination fully eliminated due to the use of the pulsed current, which breaks the surface oxides. Many of the researchers pointed that the temperature range for consolidating ODS austenitic stainless steels as 900 °C to 1000 °C during a sintering time of 15 min.

2.6. Hot Isostatic Pressing (HIP)

Hot isostatic process (HIP) is a P/M process, developed for eliminating the internal voids of materials and increases the compaction density that improves the tensile properties and workability of the metals. In this process the

pressure is applied in all directions of the sample placed in the chamber. It is a well-established process in which simultaneous application of pressure and temperature for a specified time period in order to improve the mechanical properties of the metals.

The schematic representation of HIP process is shown in Fig.3f. A mixture of pre-alloyed and elemental powders in the container is subjected to high temperature under a vacuum to remove air gaps and moisture from the powder. Then the container is hipped by the application of inert gas pressure that results in the elimination of porosity and creates a strong bond between the powder particles.

Hot isostatic pressing is widely used method for consolidating the nano-oxide dispersion strengthened materials. Xu, et al., [15] studied the fabrication of ODS austenitic stainless steels by hot isostatic process. The powders of ODS austenitic composition were carried out at a temperature of 1150 °C and a pressure of 200 MPa for 3 hours. The hipped samples showed near-net-shaped cross-section and uniform distribution of oxide particles. The tensile strength also greatly enhanced at both room temperature and elevated temperature compared to conventional austenitic steels.

3. MICROSTRUCTURE

The microstructure of oxide dispersion

strengthened austenitic steels manufactured by others through different consolidation processes are shown in Fig.4. The microstructural features of ODS austenitic steels developed by different consolidation processes are discussed here.

3.1. Conventional Sintering

Kumar, et al., [11] studied the development of ODS austenitic steels by mechanical alloying followed by conventional sintering. The consolidation was carried out at 1150 °C for one hour in argon atmosphere. He reported that the sintered part microstructure consists of ferrite and austenite phase. And also the maximum sintered density achieved by CS was 89 % of theoretical density. He concluded that the CS sintered samples showed homogenous dispersion of oxide materials (Y_2O_3) in the austenite matrix.

3.2. Microwave Sintering

Annamalai, et al., [12] compared the microwave sintering with conventional sintering during the consolidation of Y_2O_3 dispersed austenitic steels. He reported that the microstructure of samples consolidated by both the processes contains residual porosity having irregular grains, but the microstructure of MS processed sample contain more refined grains compared to the CS process (Fig.4a & b). The density and hardness of MS sintered sample increases compared to the density of CS process.

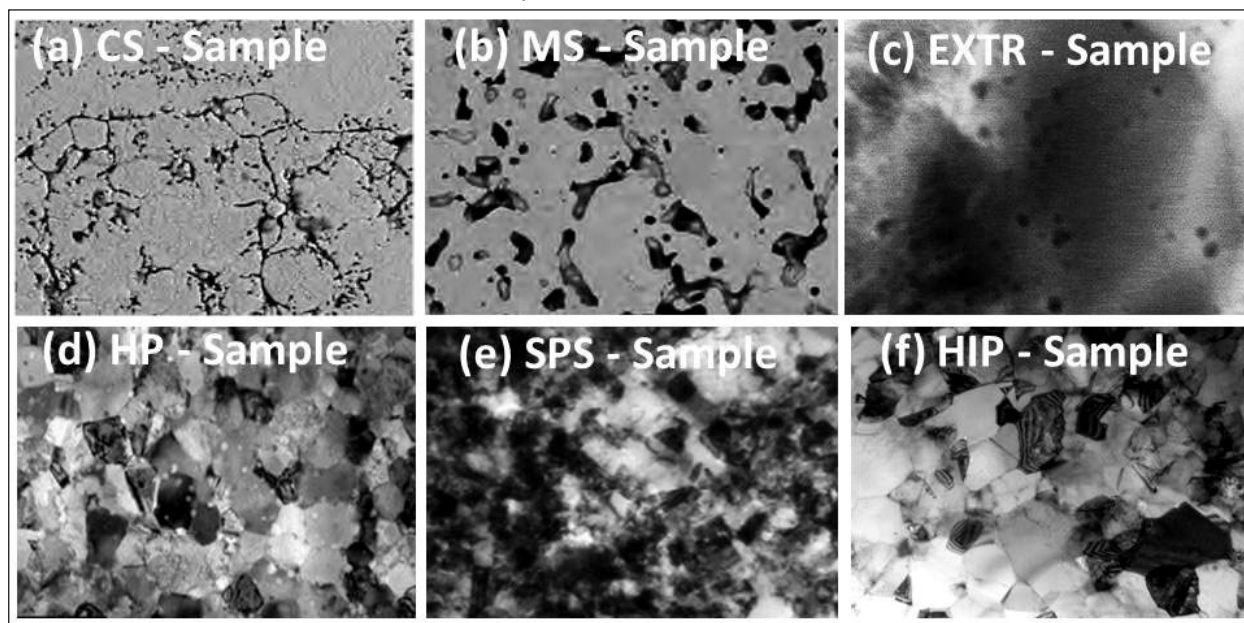


Fig 4. Microstructure of ODS Austenitic Steel by (a) CS [12], (b) MS [12], (c) EXTR [13], (d) HP [16], (e) SPS [17] and (f) HIP [4]

3.3. Extrusion

Graning, et al., [13] analyzed the mechanical properties of ODS austenitic stainless steels by hot rolling and extrusion. The microstructure of extruded sample contains more number of oxide precipitates (Fig.4c). He reported that the grain size of extruded part is fine and small compared to hot rolled part. The higher mechanical properties were achieved by hot rolling process compared to extrusion process due to very fine precipitates appeared in the microstructure of rolled sample.

Oka, et al., [18] studied the morphology of oxide particles by hot extrusion followed by heat treatment at 1373 K for one hour. He pointed that most of the oxide particles were complex oxides of faceted shape with fine size. K. Suresh, et al., [19] reported the microstructural characteristics of ODS austenitic steels by hot extrusion followed by solution treatment. He pointed that the solutionizing sample contains only austenite phase with fine equi-axed grains and no other unwanted phases in the microstructure. The solution strengthening mechanism helps to improve the properties of extruded sample.

3.4. Hot Pressing

Susila, et al., [16] studied the consolidation of ODS austenitic steels by vacuum hot pressing at the 900 °C with a pressure of 200 MPa. The nano-crystalline structure with equi-axed grains was observed in the microstructure of hot pressed samples (Fig.4d). The grain size of ODS alloy is slightly smaller than conventional steel obtained by the same process. This clearly indicates that HP process influences the grain size. They concluded that the dispersion of nano sized oxide particles are more uniform by this hot pressing process.

3.5. Spark Plasma Sintering

Mori, et al., [17] fabricated the ODS austenitic steels through the spark plasma sintering. The SPS was conducted at different temperatures of 950 °C and 1000 °C for 15 min. The ODS steel compacted at higher temperature facilitates full densification (above 99 % of theoretical density) than low temperature compacted steel. High dislocation density (Fig.4e) was obtained by the low temperature sintered sample. He concluded that the sintering time influences the volume density of nano-oxide precipitates appeared in the microstructure. And also the size of the

grain increases with an increase in the sintering temperature.

3.6. Hot Isostatic Pressing

Zhou et al., [4] fabricate the ODS austenitic steel by HIP process. The equi-axed grains (Fig.4f) of 300 nm and different kinds of dispersoids were obtained by this process. He identified the precipitate of coarse size (above 300 nm) with a composition of TiN. The properties were greatly improved by this consolidation process. Wang, et al., [20] studied the structural characterization different compositions of ODS austenitic steel powders during the HIP process. The powders after consolidation showed nano-scale grain size of around 150 to 300 nm.

Wang, et al., [21] reported the behavior of the HIPped sample after plastic deformation, including forging and hot rolling. The microstructure is more uniform after the deformation process. Moreover, the ductility increases with the deformation process and at the same time there is a little decrease in the strength of the sample was observed.

4. NANO-OXIDE PARTICLES

Suresh, et al., [19] pointed that the high temperature stability of ODS steels mainly depends on the size of oxide precipitates. The Nano-oxide Precipitates reported by different researchers through various consolidation processes are shown in Fig.5a, b, c & d. It is clearly indicating that the consolidation process significantly changes the size of nano-oxide precipitates. The precipitates obtained by HIP are very fine when compared to other processes such as extrusion, hot pressing and spark plasma sintering. The size of the precipitates for extrusion and hot pressing are shown in 100 nm scale. Whereas, the precipitates obtained by spark plasma sintering process showed in 50 nm scale. However, the precipitation of oxides by hot isostatic pressing shown in 20 nm scales. This clearly indicates that HIP is an effective process for obtaining very fine nano-oxide precipitates in the microstructure of ODS austenitic steels.

5. MECHANICAL PROPERTIES

The high temperature tensile strength of ODS austenitic steels are very much essential while operating these materials in nuclear industries. As

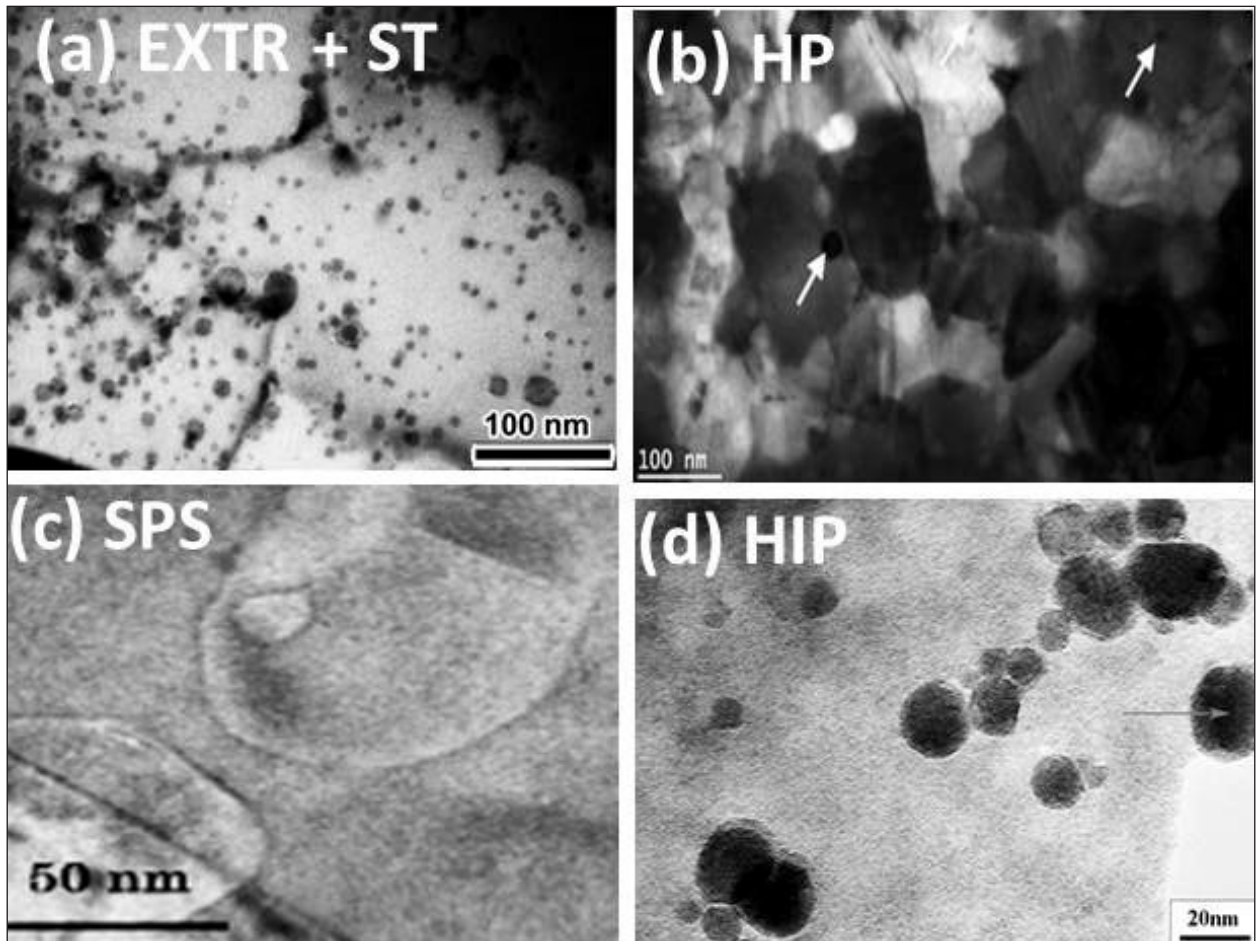


Fig 5. Nano-oxide Precipitates by (a) EXTR + ST [19], (b) HP [16], (c) SPS [17] and (d) HIP [22]

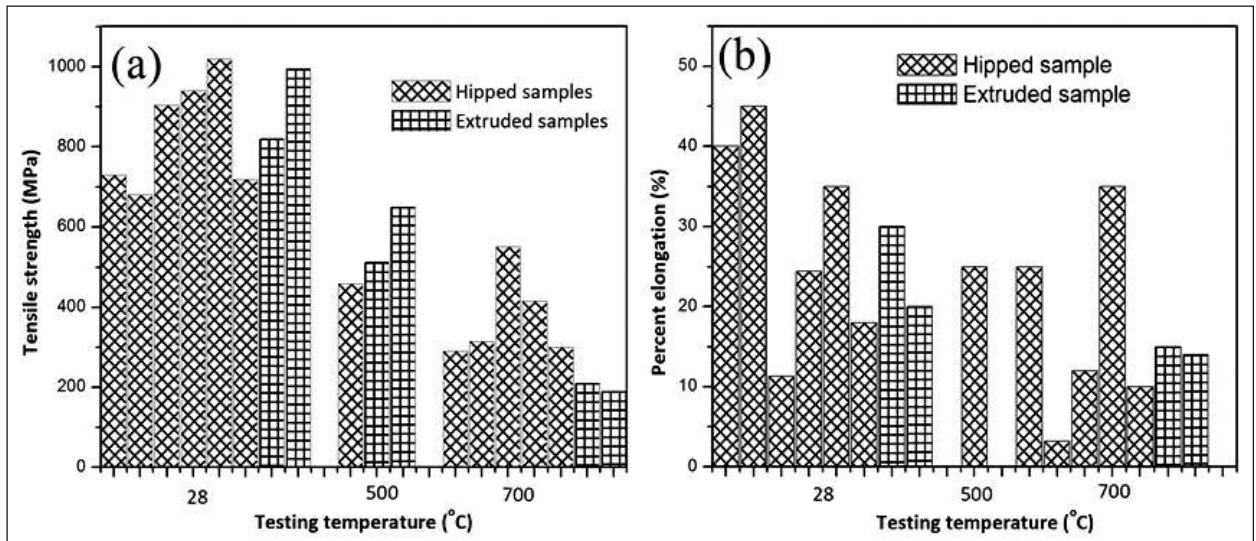


Fig 6. Mechanical Properties of ODS Austenitic Steels Reported by Researchers

per the availability of literature, the mechanical properties of ODS austenitic steels are listed in Fig.6a & b. The samples consolidated by hot isostatic pressing exhibited a maximum tensile strength of 1020 MPa at room temperature. This value is 3

times higher than conventional austenitic steels. However, the high temperature tensile strength obtained by HIP was around 600 MPa. Whereas, the highest tensile strength obtained by extrusion at room temperature and high temperature was

970 MPa and 200 MPa. These values are less than the values obtained by HIP process.

The elongation values of ODS austenitic steels developed by others are shown in Fig.6b. It is noted that the maximum elongation of ODS austenitic steel by HIP was 45% at room temperature. Moreover, the elongation of samples at high temperature (700 °C) was 33 %. On the other hand, the highest elongation achieved by extrusion at room temperature and high temperature (700 °C) was 33 % and 15 %. These values are less when compared to hipped samples.

SUMMARY

Oxide dispersion strengthened austenitic steels developed by different fabrication methods are summarized. The consolidation process for fabricating the ODS steels are reported in detail. The effect of the consolidation process on grain size, grain distribution and presence of nano-crystalline structure are discussed in detail. In addition to this the appearance of Nano-oxide dispersion strengthened precipitates with respect to manufacturing method also discussed. The mechanical properties such as tensile strength and elongation mainly depend on the consolidation process used.

The following points to be noted after a detailed review of ODS austenitic steels.

1. Grain coarsening and undesired phases were observed in the conventional sintering and microwave sintering processed ODS austenitic steels.
2. The nano-crystalline structure was obtained from the samples consolidated by HP, SPS and HIP processes.
3. The HIP processed samples showed Nano-oxide precipitates of very fine size
4. The mechanical properties of ODS austenitic steels were substantially enhanced by HIP and extrusion process.
5. The temperature strength is stable from room temperature to 700 °C for the ODS austenitic consolidated by HIP.
6. The ODS austenitic steels processed by conventional methods needs solution treatment to eliminate the unnecessary phases.

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