# A Review on Sintered Nickel based Alloys

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*Abstract* — **This paper presents a review on sintered nickel based alloys. Nickel-based superalloys are an unusual group of metallic materials, showing an extraordinary combination of high temperature strength, toughness and surface stability in corrosive or oxidative environment. Therefore, these alloys play an extremely important role in gas turbine engines, aircraft, marine, nuclear reactors, steam power plants, petrochemical equipment and other high-temperature applications. Their resistance to temperature is achieved through a mixture of dispersion hardening, precipitation hardening and solid solution strengthening. To overcome or reduce probability of segregations, difficulties of forming and machining, often associated with or present in cast ingots, powder metallurgy has been of great consideration recently. In addition, powder metallurgy enables the alloying with refractory elements for high temperature strength.**

*Index Terms*— **Sintered nickel based alloys, Microstructure, Powder metallurgy.**

## I. INTRODUCTION

The application of novel high temperature alloys in aerospace, automotive, and power industry have seen increased with recent developments. One such material that meets the need of resisting mechanical loads under high operating temperatures is nickel alloys (Jovanović *et al.*, 2011; Singh, 2016). Their temperature resistance is achieved through a mixture of dispersion hardening, precipitation hardening and solid solution strengthening (Zacherl *et al.*, 2012; Singh, 2016). As compared to any other commercially available materials, nickel-base superalloys can be used to a higher fraction of their melting points (Singh, 2016). These materials contain larger volume fractions of strengthening $\gamma'$ precipitates and refractory elements than conventional superalloys, thereby offering higher strength at high temperatures (Akca and Gursel, 2015). However, despite their superior properties, nickel alloys are being reported by Yamanoglu *et al.,* (2014), as one of the most difficult to machine materials in order to satisfy production requirement. In addition, these alloys have become more complex with more alloying elements designed for higher strengths and deforming the alloys by mechanical methods with the cast and wrought route has become more difficult (Mignanelli, 2012; Singh, 2016). Therefore, many studies have been conducted recently on the machinability of nickel based alloys. The fabrication of near net shape components by powder metallurgy (PM) offers important advantages in terms of material yield and number of production stages (Ozgun *et al*., 2013).

Furthermore, PM of superalloys has attracted great attention in recent years (Ozgun *et al.*, 2013). This is from the fact that PM overcome problems that adversely affect part-related characteristics and increase cost of production encountered in the production of the superalloys by traditional methods such as segregation, difficulties of forming and machining (Ezugwu *et al.*, 1999; Pusavec *et al.*, 2011; Ozgun *et al.*, 2013). The process (PM) is reported (Lall, and Starr, unknown year) to manufacture components that can result in highly ductile (>30% elongation) or very strong (>1500 MPa UTS) products. This wide range of properties is attributed to the relative ease with which compositions can be tailored and that various processing parameters (compaction pressures, sintering time and temperatures, post-sinter operations, heat treatment, surface modifications, etc.) can be varied for the optimum microstructural evolutions. Spark plasma sintering (SPS) is a novel powder metallurgy sintering technique that is reported to be more effective as compared to the conventional sintering techniques (Garcia *et al.*, 2010). The duration of the high-temperature stage is reduced, thus, lowering the overall sintering time. Sintering is conducted at comparatively lower temperatures with shorter dwell times reducing the threat of vaporization and resulting in dense, fine grain structures exhibiting high strength (Luke, 2010; Yamanoglu *et al.*, 2014; Shongwe *et al.*, 2015;). Detailed information on this technology (SPS) are presented in Refs (Shongwe *et al.*, 2015; Makena *et al.*, 2017a).

In most cases nonreactive environments such as vacuum or an inert gas are used to avoid contaminations. Characteristics of the starting powders, such as degree of oxidation, segregation, morphology, and purity, are generally of significance prior to sintering. During sintering, it is reported by Davis, (2000), that loosely packed beds or

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compacted preforms of carbonyl nickel powders do not densify at <600 °C (1110 °F). However, considerable strengthening of compacted materials occurs at <600 °C (1110 °F) due to surface diffusion. As the temperature increases above 600 °C (1110 °F), volume diffusion increases. At ~950 °C (1740 °F), filaments in the porous structures become smooth and broadened, while in consolidated structures the pores become rounded. To attain adequate strength without excessive loss of porosity, sintering temperatures between 950 and 1000 °C (1740 and 1830 °F) are optimum.

It is reported by Pasebani *et al.,* (2015), that conventional nickel based alloys are not stable at high temperatures. This is attributed to the coarsening or dissolution of the second phase particles. Often, the remedial actions are associated with homogeneous dispersion of nanometric stable oxide particles in the matrix of nickel based alloys. Nickel based oxide dispersion strengthened ODS alloys, reinforced by homogeneously dispersed nano-particles (usually $Y_2O_3$), are quite stable during high temperature applications in excess of 1000 °C. Homogeneous dispersion can act as effective barriers against dislocation motion and improve high temperature mechanical properties including creep strength.

## II. LITERATURE SURVEY

**Yamanoglu *et al.*, (2014)** characterised nickel alloy powders processed by spark plasma sintering. Experiments were performed between 700 and 750$^o$C temperature range under 50 MPa pressure with holding times from 5 to 10 min. In addition to these main spark plasma sintering parameters three different heating rates ranging from 100 to 235$^o$C/min and two different particle size ranges (75–106 mm narrow size distribution and 245 mm wide size distribution) were used for the experiments. After sintering, the sliding wear behaviour of the samples was investigated. The results revealed that the density of the material increased with raising the sintering temperature and holding time. However, heating rate and particle size also played an important role in the densification and these parameters were investigated in detail.

**Shongwe *et al.*, (2015)** studied the effect of sintering temperature on the microstructure of hybrid hot press spark plasma sintered Fe-30%Ni alloy. It was observed that the relative density, microhardness and fracture morphology depend on the sintering temperature which also affects the microstructure. The densification and grain size of the alloys increased with increasing sintering temperature, facilitating necking of grains. In the case of the sintering temperature at 1230$^o$C, a relative density of 98.7% and maximum grain size of around 200 mm were obtained, and the maximum microhardness of 284 $Hv_{1.0}$, and the microhardness indentations revealed pin cushioning indicating better sintering. Microhardness indentations at 1100$^o$C and below were reported to be characterized by barreling, indicating poor densification and/or microhardness. The fracture type changing from intergranular fracture to transgranular fracture was an indication of improved consolidation of the Fe-30%Ni alloy with increasing sintering temperature.

**Makena *et al.*, (2017a)** used SPS technique to fabricate Ni50Fe alloys. Powders were sintered at various temperatures, pressure and holding times ranging between 900 and 1100$^o$C, 40-50 MPa and 5-10 min, respectively. Microstructural characterisation was carried out using scanning electron microscopy (SEM). Archimedes' method for density measurements, vickers microhardness, and tribometer were used to study the effect of various sintering parameters on the densification, electrochemical properties in 1 M $H_2SO_4$ solution, coefficient of friction (CoF) and dry sliding wear resistance. Within the range of parameters used, the highest values of the characterized mechanical properties (microhardness) and corrosion resistance were obtained at a sintering temperature of 900$^o$C, sintering time of 10 min, and applied pressure of 50 MPa. Highest values of 235 $H_v$ and 98.55% were obtained for microhardness and the relative density for 900$^o$C and 1100$^o$C, respectively. Sintering of an alloy at higher temperatures (> 900$^o$C) reduced its resistance to sulphuric acid attack. The influence of varying both sintering time and pressure on an acid attack was found to be negligible. Increasing temperature was also found to be reducing corrosion resistance but improving alloy wear resistance against the tungsten carbide counter body. Fractography studies indicated the dominant fracture micromechanism as ductile fracture of sintering necks. The main wear mechanism of Ni-50Fe alloy was related to the simultaneous actions of ductile and brittle friction mechanism with the existence of abrasion and delamination of worn surfaces

**Garcia *et al.*, (2010)** used spark plasma sintering technique to densify commercial NiCoCrAlY-Ta alloy. Such powder was sintered at temperatures ranging between 900 and 1050 °C to yield various microstructures. A decrease in porosity was clearly observed with increasing sintering treatment temperature. An analysis of the hardness in the sintered samples by SPS, shows a rise in Vickers hardness with respect to the sintering temperature. However, major hardness resistance was obtained at 1000 °C. In contrast, at temperature of 1050 °C it was observed a diminution of hardness and an increase of the porosity that could be associated with the start of the melting components.

**Luke (2010)** compared the microstructure of nickel produced by SPS at varying ramp rate, temperature, dwell time, and applied pressure, and nickel produced at varying time and temperature by conventional sintering (CS) technique. Material properties that were compared included: density, hardness, porosity, average grain size, and grain boundary character. Evaluations of the activation energy of sintering, plastic flow, and modeling using a hot-press equation of SPS nickel were also reported in order to elucidate the mechanism of sintering. The specimens processed by SPS reached higher density at lower temperatures than those processed by CS techniques. The hardness of SPS nickel was observed to be linearly related to the density. The hardness and porosity results also indicated that radial temperature gradients do exist during SPS processing of nickel. The average grain sizes did not change significantly with SPS process conditions except at high temperatures or for long dwell times. Approximations of the grain growth rate of SPS nickel were observed to be higher than for CS nickel. The grain boundary character is stable during both SPS and CS processing, with significant increases in the Σ3 and special fractions only occurring at the longest times and highest temperatures. The activation energy of sintering indicated that SPS nickel densifies by diffusional processes. A hot-press model was also used to help identify the sintering mechanism, but the model was insufficient to explain the SPS process. The microstructural evolution of nickel was different during processing with SPS and with CS techniques.

**Rodrigues *et al.*, (2014)** investigated the effects of compaction pressure on the final microstructure and density of Fe50Ni Alloy. Cylindrical samples with around 6.5g/cm$^3$ of green density were vacuum sintered at 1220$^o$C. There was no effect of the re-compaction pressure on the final densities; Higher densities were obtained for samples treated at higher temperature and longer time; Due to the presence of unalloyed iron, the corrosion resistance for under sintered samples was quite poor; Alloying started during heating step and for a sample sintered at 1280$^o$C the microstructure was austenitic.

**Yamanoglu *et al.*, (2014)** consolidated nickel alloy powders by spark plasma sintering. Experiments were performed between 700 and 750$^o$C temperature range under 50 MPa pressure with holding times from 5 to 10 min. In addition to these main spark plasma sintering parameters three different heating rates ranging from 100 to 235$^o$C/min were used for the experiments. The results revealed that the density of the material increased with raising the sintering temperature and holding time. Optimized sintering conditions were obtained as the sintering temperature of 750$^o$C, a dwell time of 5 min, a heating rate of 100$^o$C/min. The final relative density for these parameters was 99.03%. The wear resistance of the material was reported to increase with increasing density. The samples showed increased resistance against to ceramic ball (alumina). Depending on the hardness of the steel and ceramic counterface the wear mechanism changed from abrasive to delamination wear.

**Makena *et al.*, (2017b)** investigated the effect of sintering temperature on mechanically milled Ni and Iron, respectively, with Co additions (Ni40Fe10Co) on the densification and microstructural evolutions. Admixed powders were sintered at various temperatures ranging between 900 - 1100°C. Materials characterization of powders and sintered compacts were done using scanning electron microscopy (SEM), Optical microscope and X-ray diffraction (XRD). Milling of Ni for 20hrs produced larger cold welded clusters, while Fe milling resulted in finer rod-shaped particles. The observed agglomerating behaviour of Ni during milling was associated with its ductility. With an increase in sintering temperature, no substantial detectable new phases were observed. Optical images of polished surfaces revealed that with an increasing sintering temperature the pores coalesce, become spherical and thereafter grows. This was linked with the interdiffusion/phase transformations between the ternary particles upon heating and cooling cycle during sintering.

**Ergin et al. (2015),** synthesised Inconel 718 nickel based superalloy in an electric current activated sintering in open air under a uniaxial pressure of 300 MPa using Al, Ni, Fe, Cr, Ti, Co, Fe-Nb and Fe-Mo powders. Optical and SEM examinations showed a dense microstructure with low amount of porosity. XRD studies showed the existence of the $\gamma$ and MC type carbide (M atom is Nb, Ti combination) and $\delta$ phases. The relative density of sample measured according to Archimedes' principle was 93.7%, and the microhardness of sample was about 343.8 HV$_{0.1}$.

**Fuys JR *et al.*, (1976)** studied the physical properties of a nickel-base alloy prepared by isostatic pressing and sintering of the powdered metal. The particle sizes of the powders used to make the samples varied from -80/ +200 mesh to -325 mesh. The compaction pressure varied from 138 to 414 MN/m$^2$ and the sintering temperature varied from 1150 to 1250 $^o$C. The shrinkage during processing, the porosity, tensile strength, yield strength, elongation, and elastic modulus were used to characterize the samples. The strength of the samples generally increased with decreasing particle size of the powder and increasing compaction pressure and sintering temperatures. The porosity and strength, therefore, could be varied over a wide range by controlling the various parameters. The properties of the samples prepared by powder metallurgy were compared with those of the cast alloy and compact bone. Conditions can be selected that will yield equivalent or better properties by powder metallurgy than by casting.

**Pasebani *et al.*, (2015)** investigated the oxide dispersion strengthened nickel based alloys via spark plasma sintering. The oxide dispersion strengthened (ODS) nickel based alloys were developed via mechanical milling and spark plasma sintering (SPS) of Ni–20Cr powder with additional dispersion of 1.2wt% $Y_2O_3$ powder. Furthermore, 5wt%$Al_2O_3$ was added to Ni–20Cr–1.2$Y_2O_3$ to provide composite strengthening in the ODS alloy. The effects of milling times, sintering temperature, and sintering dwell time were investigated on both mechanical properties and microstructural evolution. A high number of annealing twins was observed in the sintered microstructure for all the milling times. However, longer milling time contributed to improved hardness and narrower twin width in the consolidated alloys. Higher sintering temperature led to higher fraction of recrystallized grains, improved density and hardness. Adding 1.2wt%$Y_2O_3$ to Ni–20Cr matrix significantly reduced the grain size due to dispersion strengthening effect of $Y_2O_3$ particles in controlling the grain boundary mobility and recrystallization phenomena. The strengthening mechanisms at room temperature were quantified based on both experimental and analytical calculations with a good agreement. A high compression yield stress obtained at 800$^o$C for Ni–20Cr–1.2$Y_2O_3$–5$Al_2O_3$ alloy was attributed to a combined effect of dispersion and composite strengthening.

**Chang *et al.*, (2013)** studied the improvement of mechanical and electrical properties on the sintered Ni50 mass% Cr alloys by HIP treatment. The study included investigation of Ni50 mass% Cr alloys produced by the vacuum sintering and hot isostatic pressing (HIP) of powder metallurgy technology. The experimental results showed that the relative density of Ni50 mass% Cr alloys reached 98.67%, the apparent porosity decreased to 1.33%, transverse rupture strength (TRS) increased to 454.29MPa and electrical resistivity decreased to 4.284 x $10^{-4}$ Ω·cm after 1345°C sintering for 1 h. Meanwhile, laminar eutectic precipitations appeared in the sintered Ni50 mass% Cr alloys. In addition, the relative density increased to 99.73%, the apparent porosity decreases to 0.27% and TRS was obviously enhanced to 1181.4MPa after 1260°C 175 MPa 4 h HIP treatment. Moreover, the electrical resistivity decreased to 3.346 x $10^{-4}$ Ω·cm after the optimal HIP treatment. This study showed that the HIP process is effective in eliminating internal pores and improving the mechanical and electrical properties of the sintered Ni50 mass% Cr alloys, thus obtaining the high density and optimum properties of the sintered materials.

**Ozgun *et al.*, (2013)** investigated the Injection molding of nickel based 625 superalloy: Sintering, heat treatment, microstructure and mechanical properties. The study involved the determination of optimum sintering and thermal process parameters for Ni-based alloy 625 superalloy formed by the method of powder injection molding (PIM). Samples, formed from the feedstock by mixing the prealloyed 625 powder with a multi-component binding system, are made subject to sintering at different temperatures following the debinding process. Samples that are sintered under such conditions giving way to the highest relative density (3 h at 1300 $^{o}$C), are aged after they have been subject to solution treated thermal process. Sintered, solution treated and aged samples have been subjected to microstructural analysis and mechanical test. Mechanical tests such as hardness measurement and tensile test as well as microstructural characterization such as X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM) and elemental analysis all have shown that the aging thermal process increases strength of the material. However, it is observed that alloy 625 produced by the method of PIM is at such level to compete with the characteristics of cast alloy 625.

As reported by **Kitaguchi (2012),** the microstructure particularly the grain boundaries, was found to be controlled by the two factors. Firstly, it was the nominal chemical composition, especially the hardening precipitate participants. Secondly, heat treatment has a profound influence of microstructure. The damage tolerance properties are also concerned with the environmentally assisted crack propagation along grain boundaries, which is essentially the oxidation assisted crack propagation in this study. In general, chromium oxide ($Cr_2O_3$) has been regarded as a healing agent of the oxidation process in Ni alloys. It was further reported that the oxidation sequence followed the free energies for the oxides to form. Thus, for example, $Cr_2O_3$ is one of the earliest oxides to form at the crack tip.

**Zacherl *et al.*, (2012)** investigated the effects of alloying elements on the creep rate of Ni-base superalloys, and the factors entering into a secondary creep rate were calculated via first-principles calculations based on density functional theory for 26 $Ni_{31}X$ systems where X = Al, Co, Cr, Cu, Fe, Hf, Ir, Mn, Mo, Nb, Os, Pd, Pt, Re, Rh, Ru, Sc, Si, Ta, Tc, Ti, V, W, Y, Zr, and Zn. It was found that shear modulus, Young’s modulus, and roughly stacking fault energy show inverse correlation to the atomic volume of the system. In addition, the closer the alloying elements to Ni, with respect to atomic volume and atomic number, the larger the predicted shear modulus, Young’s modulus, and stacking fault energy. Diffusivity calculations shown that mid-row 5d transition metal elements, particularly Re, Os, and Ir, have the highest activation barrier for diffusion, while far-right or far-left row placement elements such as Y, Zn, and Hf, have the lowest activation energy barriers for diffusion.

An investigation on the structural stability and stress rupture properties of the existing alloy GH984 (Ni–Fe based alloy) was done by **Wang *et al.*, (2015).** It was observed that the most important changes in the alloys are γ′ coarsening, the γ′ to η transformation and the coarsening and agglomeration of grain boundary $M_{23}C_6$ during thermal exposure. The stress rupture strength was found to be slightly lower than the requirement of 700 °C A-USC. The fracture mode of creep tested specimens was intergranular fracture. Detailed analysis revealed that η phase precipitation is sensitive to Ti/Al ratio and can be suppressed by decreasing Ti/Al ratio. The coarsening behavior of γ′ phase is related to Fe content. Based on the research presented and analysis of the data, a modified alloy was developed through changes in composition.

**Nabeel (2012)** investigated the influence of alloying additions on mechanical properties and microstructure homogeneity. It was reported that an increase in hardness and strength occurs with increasing sintering time, which is more prominent in Ni containing compositions. Individual addition of Mo to Fe-Ni system had a negative effect on uniformity of Ni. However, the results showed that the Ni distribution was improved by Mo when added to Fe-Ni-C. The phenomenon is attributed to decrease in chemical potential of carbon due to Mo addition. It was further observed that the experimental diffusion rate is faster than idealized volume diffusion, which indicates that grain boundary diffusion and surface diffusion are important diffusion mechanisms for distribution of elemental additives during sintering. Individually all these alloying elements enhanced the hardness and strength. When all the alloying elements were present at the same time, the hardness and strength increased dramatically. It was then concluded that microstructural modifications due to alloying elements have important influence on the mechanical properties. Other than the individual effects of each alloying element, the interaction between alloying additions have major contribution in final properties.

The structure and mechanical properties of $Ni_3Al$ intermetallic compound were studied by **Shevtsov *et al.*, (2014).** Materials were fabricated according to different schemes, which combined mechanical alloying of Ni and Al powders, self-propagating high temperature synthesis (SHS) and spark plasma sintering (SPS). Relative density of all sintered samples was ~ 97 %. Microhardness of the sintered materials ranged from 6100 to 6300 MPa. SPS of 86.71 % wt. Ni and 13.29 % wt. Ni powder at 1100 $^{o}$C led to the formation of material with the highest level of tensile strength equal to 400 MPa.

As reviewed by **Akca and Gursel (2015)**, to increase the strength of polycrystalline Ni-based superalloys, levels of refractory alloying additions and γ'-forming elements have gradually increased to levels that make conventional processing routes deficient. Elements such as W, Mo, Ti, Ta, and Nb effectively strengthen the alloy but also result in severe segregation within the ingot upon solidification. Additionally, the limited ductility of the high-strength alloys renders the ingot susceptible to cracking as thermally induced stresses evolve during cooling. Powder-processing routes have been developed to overcome the difficulties associated with melt-related defects and are viable for the production of advanced high-strength polycrystalline superalloy components. Listed in Table 1 below are the compositions of some commercially available powder processed Ni-based superalloys (Akca and Gursel, 2015).

Table 1: Compositions of commercial Ni-based superalloys processed by powder techniques (wt. % bal. Ni).

| | Cr | Co | Mo | W | Nb | Al | Ti | Hf | C | B | Z |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Rene' 95 | 13.0 | 8.0 | 3.5 | 3.5 | 3.5 | 3.5 | 2.5 | _ | 0.07 | 0.01 | 0.05 |
| Rene' 88 DT | 16.0 | 13.0 | 4.0 | 4.0 | 0.7 | 2.1 | 3.7 | _ | 0.03 | 0.02 | _ |
| N18 | 11.2 | 15.6 | 6.5 | _ | _ | 4.4 | 4.4 | 0.5 | 0.02 | 0.02 | 0.03 |
| IN100 | 12.4 | 18.4 | 3.2 | _ | _ | _ | _ | _ | _ | _ | _ |

## III. Conclusion

The inferences that can be drawn based on the literature review given above are:

a) The use of powder metallurgy as a processing route expunge the necessity of costly machining steps and also enables the addition of refractory elements designed for higher strengths.

b) Sintered nickel alloys can compete with cast alloys, and composite strengthening is possible through mechanical alloying and sintering.

c) Near full densified nickel alloys can be effectively produced. During sintering, an increase in sintering temperature, pressing pressure and holding time enhances densification significantly.

d) Two major factors that affect microstructural evolutions in nickel based alloys are nominal compositions and heat treatment.

e) Sintered nickel-base materials enjoy wide commercial usage due to their unique properties, such as corrosion resistance, wear resistance, mechanical strength at low and elevated temperatures, thermal expansion, electrical conductivity, and magnetic permeability. Their resistance to temperature is achieved through a mixture of dispersion hardening, precipitation hardening and solid solution strengthening.

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