

Article

Fatigue Behavior of Alloy Steels Sintered from Pre-Alloyed and Diffusion-Bonding Alloyed Powders

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Abstract: Porosity and phases are considered to be two key factors for the fatigue performance of powder metallurgy steels. In this paper, the fatigue strengths of the alloy steels sintered from two typical types of powders, pre-alloyed Fe-Cr-Mo (Astaloy CrM), and diffusion-bonding alloyed Fe-Cu-Mo-Ni (Distaloy AE), were comparatively analyzed in view of the geometry of porosity, the phases constitution, and fractography of fracture. Different modes of fatigue fracture were distinguished between the two materials. Namely, a trans-particle fracture is predominant in the Distaloy AE steel due to the heterogeneous phases which consist of soft phases in powder interior and hard phases along powder borders. In contrast, the fatigue fracture of the Astaloy CrM steel with a homogeneous mono-phase of martensite is characterized by an inter-particle fracture at the sintering necks. Moreover, the fatigue endurance limit of the Distaloy AE steel was not pronouncedly improved by increasing sintering temperature in comparison with the Astaloy CrM steel. This was attributed to the softening of the network constructed by martensite at sintering necks. A modified Murakami model which considers micro-scale defect and micro-hardness is effective to predict the fatigue performance of the alloy steels sintered from pre-alloyed and diffusion-bonding alloyed powders, respectively.



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Keywords: powder metallurgy; alloy steel; porosity; fatigue

1. Introduction

Powder metallurgy (PM) is a cost-effective technology to consolidate metal elements or alloyed powders into dense bulk materials [1] and has been widely used in the mass production of automotive components of alloy steels, e.g., synchronizer hubs, connection rods, and gears, which desires not only good performance of sustaining a heavy load of cyclic stress but also light-weight and fuel efficiency. For automotive components under cyclic load, fatigue fracture occurs commonly to cause their failure. Previous studies indicated that the fatigue cracking behavior of the alloys strongly depended on the size and morphology of porosity and phases which were formed with different types of alloy elements and processing methods [2,3]. These technical factors, e.g., porosity, phase, alloying method, and consolidation temperatures, must be considered to understand fatigue behavior and help to design fatigue performance. The porosity would be unavoidably present in the microstructure of steel alloys manufactured by powder metallurgy and plays an important role in the fatigue performance of alloys because fatigue cracks are most likely to be originated from pores where stresses are concentrated [4,5]. By reducing the size and amount of pores, especially the large ones, the fatigue strength of the PM steel alloys could be effectively improved [6,7].

The fatigue strength of alloy steels can be greatly affected by the phase constitution that could be composed of multiple phases, e.g., pearlite, bainite, martensite, and austenite. The origin of cracks is fostered in the soft austenite phase, but the cracks can propagate fast

in the hard and brittle martensite phase [8,9]. For example, the speed of crack growth in the nickel-enriched phase is three times higher than that in the coarse pearlite phase [10]. However, it was reported that the fatigue strength can be improved when the nickel-enriched austenite phase acts as an obstacle to crack propagation [11], and also by the martensite phase formed under strain from ductile austenite at sintering necks. In this case, energy was dissipated by phase transformation instead of being consumed for the origination and propagation of micro-cracks [12,13]. Further improvement of fatigue strength can be achieved by suitable heat treatments [14].

The methods of alloying primary elemental powders, e.g., pre-alloying, diffusion-bonding alloying, or admixed alloys powders, are crucial to achieving good fatigue performance of powder metallurgy steels. The concept of diffusion-bonding alloying derives from the intention to eliminate element segregation and obtain a heterogeneous microstructure formed by blending fine powders of alloying elements with coarse powders of iron. Instead, a homogeneous microstructure is easily formed by the pre-alloying method, resulting in poorer compressibility than the heterogeneous microstructure formed by the diffusion-bonding alloying technique [15]. Pre-alloyed powders with low-cost chromium and manganese give an excellent hardenability, although sensitive to oxygen, which is very attractive to manufacturing automotive components bearing heavy loads [16–18].

By now, the studies on elucidating the differences in the effects of alloying technologies on fatigue resistance performance are very limited in the literature, although there are already some investigations on the roles played by porosity and phases in fatigue cracking behavior of the sintered powders of diffusion-bonding alloyed (Distaloy AE), which exhibited a two-step crack growth and bifurcation [19], as well as the arrest or circumvention of fatigue cracks in the nickel-enriched region [20,21], or pre-alloyed (Astaloy CrM), which was characterized by an exponent of the Paris law at different sintering temperatures [22]. In this paper, the effects of porosity and the constituting phases on the fatigue strength will be analyzed for the alloy steels consolidated from two types of powders, i.e., pre-alloyed with chromium and molybdenum (Astaloy CrM), and diffusion-bonding alloyed with nickel, molybdenum, and copper (Distaloy AE). The relationships among fatigue strength, porosity, and the phase constitution, together with the origin and propagation of cracks were clarified. A quantitative relationship between porosity/phases and fatigue strength was established by a modified Murakami model.

2. Materials and Methods

Alloy steels were consolidated from two types of base powders, i.e., pre-alloyed powders (Astaloy CrM, Höganäs AB, Höganäs, Sweden) containing 3 wt.% chromium and 0.5 wt.% molybdenum, and diffusion-bonding alloyed powders (Distaloy AE, Höganäs AB, Höganäs, Sweden) by diffusion bonding pure iron particles with 4 wt.% nickel, 1.5 wt.% copper, and 0.5 wt.% molybdenum. The chemical compositions of both materials are shown in Table 1.

Table 1. Chemical compositions of materials.

Alloy Steel	Cr (wt.%)	Mo (wt.%)	Cu (wt.%)	Ni (wt.%)	Fe (wt.%)
Astaloy CrM	3.0	0.5	-	-	Bal.
Distaloy AE	-	0.5	1.5	4.0	Bal.

Graphite (natural graphite UF4) with fractions ranging from 0.35 wt.% to 0.85 wt.% were added to both materials to investigate the effects of microstructures on fatigue properties. The materials were compacted in a hydraulic CNC press to prepare standard bar specimens for fatigue tests (ISO 3928:1999) with a density of around 7.0 g/cm³. Then, the compacts were sintered at 1120 °C for 30 min in the atmosphere of mixed gases consisting of 90 vol.% H₂ and 10 vol.% N₂. The majority of the sintered samples (Distaloy AE) were cooled at a low rate of 0.8 °C/s, whereas the sinter-hardened steel (Astaloy CrM) was produced at a fast cooling rate of 2.5 °C/s. Further, the material with 0.5 wt.% graphite

was selected to investigate the effects of porosity on alloy fatigue performance, compacts with different compacting densities ranging from 6.70 g/cm³ to 7.20 g/cm³ were designed, which were subsequently sintered at 1120 °C for 30 min.

The bars with a density of 7.0 g/cm³ were sintered at 1120 °C and 1250 °C, respectively, and then cooled at 0.8 and 2.5 °C/s to study the effects of consolidation temperature. Finally, all sinter-hardened bars (Astaloy CrM) were tempered at 200 °C for 60 min in the air to eliminate any residual stress caused by fast cooling.

The fatigue tests of the sintered bars were undertaken by a designed apparatus from Höganäs AB inducing the fatigue by a displacement controllable planar bending with a frequency of 25–30 Hz and a load ratio $R = -1$. The corners of every testing specimen were carefully polished to remove burr. The fatigue tests were terminated once the failure of the testing bar was verified with the increase in gauge length by 2.5%. Testing data were collected by the staircase method with a run-out limit by 2×10^7 cycles. The analysis of the fatigue data is adaptable for steels having an endurance limit according to standard MPIF standard 56. The staircase analysis is useful to determine a statistical mean value of the endurance limit σ_A .

The density of the sintered sample was measured by the Archimedes method. The size and roundness of pores were estimated by a light optical microscope (Leica DM6 M, Leica Camera AG, Weztlar, Germany) equipped with a Leica LAS software, LAS 4.13, Leica Camera AG, Weztlar, Germany. Metallographic features of phases and the fracture surfaces were observed in an etched sample by scanning electron microscopy (SEM, HITACHI S-3400N, SE, at 10 kV, Hitachi High-tech Corporation, Tokyo, Japan). The etchant is 4% Picral for Distaloy AE and 1–2% Nital for Distaloy CrM. The contents of carbon and oxygen of the sintered bars were examined by a Leco analyzer (CS-844&TC-500, LECO Corporation, St. Joseph, MI, USA). Micro-hardness of the sintered samples was determined as the average value from 7 hardness tests by a Vickers micro-hardness tester (KB 30S) under a load of 10 g for a dwelling time of 14 s.

3. Results and Discussion

3.1. Effects of Porosity on Fatigue Strength

The porosity in powder metallurgy steels could greatly influence alloy fatigue performance under cyclic loading conditions because stresses are most likely concentrated in the vicinity of acute edges of pores with irregular shapes, where plastic deformation and micro-cracks are originated. Figure 1 shows the fatigue strength obtained at various levels of sintered density in both the steels made from diffusion-bonding alloyed powders (Distaloy AE) and pre-alloyed powders (Astaloy CrM), respectively. Obviously, the fatigue strength of both sintered alloy steels was improved with an increase in compacting density, and the fatigue resistance of Astaloy CrM was better than that of Distaloy AE. In addition, the efficiency of strengthening by increasing the density (the slope of linear correlation is similar and the R^2 is more than 0.96 for both lines) in the Astaloy CrM steel is similar to that in the Distaloy AE steel. Figure 2 presents the morphology of porosity and sintering necks in the Astaloy CrM steels with different consolidation densities. The higher degree of consolidation density, the lower the porosity and the greater the development of sintering necks would be, which means the surface area of the contact between adjacent particles is augmented. As a result, the absorption of more energy is the prerequisite for destroying the bigger sintering necks as the channel of crack propagation. Undoubtedly, the elimination of porosity is decisive to prevent fatigue failure. Therefore, porosity is a main deciding factor of fatigue performance in both types of powder metallurgy steels once their phases are identical.

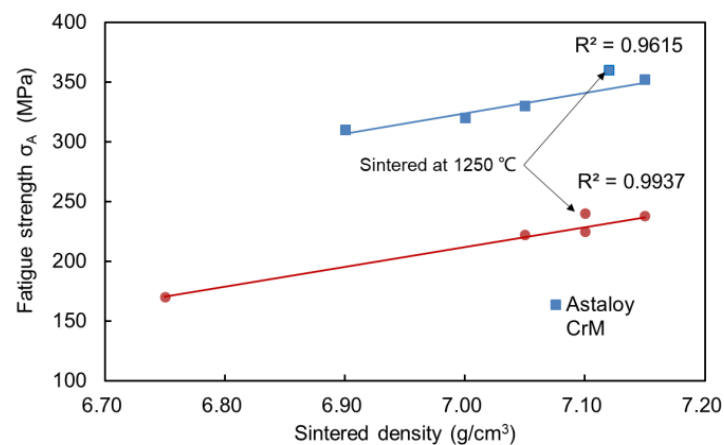


Figure 1. Fatigue endurance limit (σ_A) of alloy steels as a function of density. The alloy steels were sintered at 1120 °C, then cooled at 2–3 °C/s (for Astaloy CrM), or naturally cooled (for Distaloy AE).

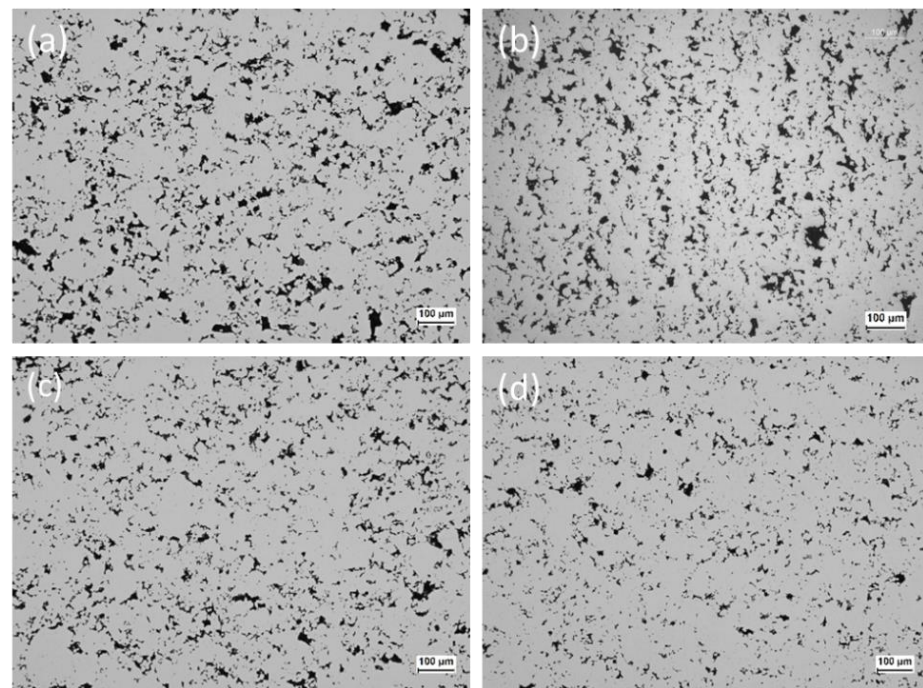


Figure 2. The morphology of porosity and sintering necks at different levels of density: (a) 6.75 g/cm³ (porosity 14.23%), (b) 7.00 g/cm³ (porosity 11.05%), (c) 7.05 g/cm³ (porosity 10.4%), and (d) 7.15 g/cm³ (porosity 9.15%).

Moreover, rising sintering temperature resulted in a better fatigue strength for both types of alloyed materials, where the improvement from 320 MPa to 360 MPa was observed for Astaloy CrM and from 222 MPa to 240 MPa for Distaloy AE when the sintering temperature was increased from 1120 °C to 1250 °C. It can be noted the efficiency of increasing sintering temperature is not pronounced to improve the fatigue strength of Distaloy AE steel in comparison with Astaloy CrM.

As shown in Figure 3 and Table 2, statistical analysis with LAS image was undertaken to reveal geometric features of the porosity of the samples sintered in different temperatures. Small pores were gradually eliminated by increasing sintering temperature, as well as shrinkage of the average pore area ($\bar{A}$). Moreover, the roundness of pores was improved by rising sintering temperature. In other words, stress concentration was alleviated owing to the annihilation of acute angles of irregular shapes. As a result, fatigue strength was improved. The area of largest pores (A_{max}) residual in Astaloy CrM steel increased, whereas

it shrank in Distaloy AE steel with rising sintering temperature. Obviously, the growth of the largest pores and elimination of small pores are spontaneously driven by the promoted atom diffusion at high temperatures in pre-alloyed Astaloy CrM steel. However, the shrinkage of the largest pores could be benefited from alloying with elements such as nickel and copper, which are highly diffusive at high temperatures in the Distaloy AE steel. Overall, pores were eliminated or rounded by an increase in density and sintering temperature. Thus, a larger strain or less stress concentration critical to the origination of cracks is required, which means the exhibition of better fatigue strength.

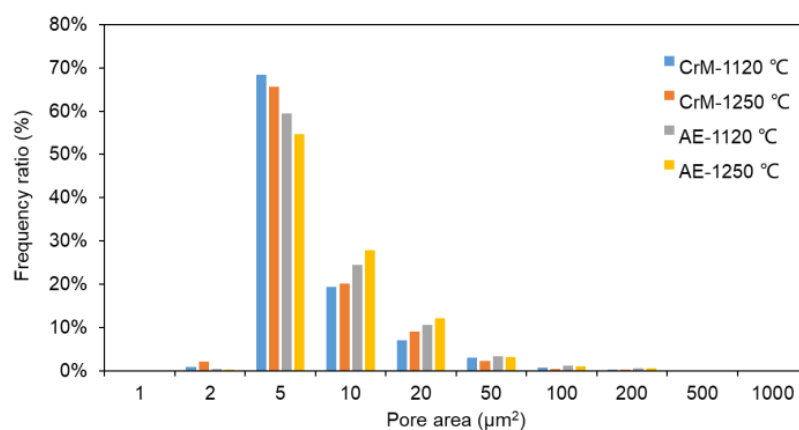


Figure 3. The size distribution of residual pores in the alloy steels sintered at 1120 and 1250 °C, respectively.

Table 2. Statistical analysis of pore size and roundness after sintering.

Sample	$\bar{A}$ (μm ²)	A_{max} (μm ²)	Average Roundness (μm)
CrM-1120	64.3	3892	2.1
CrM-1250	50.1	3940	1.8
AE-1120	77.5	4749	2.0
AE-1250	73.3	4242	1.8

3.2. Effect of Phases on Fatigue Strength

The concentration of carbon in steel is an indispensable factor in determining the type of phases and mechanical properties. Thus, Astaloy CrM and Distaloy AE steels with different carbon contents were designed, which were sintered at 1120 °C, and then cooled at a slow rate to form multi-phases, or rapidly cooled to form a pure martensite phase. Figure 4 shows fatigue strength (σ_A) obtained through the various composition of phases. When the carbon content was less than 0.5 wt.%, the Astaloy CrM steel predominantly composed of bainite has an inferior fatigue strength. The fatigue strength was significantly increased by the increase in carbon content (=0.53 wt.% and 0.56 wt.%, respectively), in slowly cooled Astaloy CrM steels, which have a large fraction of martensite (=30 vol.% and 60 vol.%, respectively). Pure martensite microstructure was obtained when the alloy is fast cooled which had the maximum fatigue strength up to 380 MPa, 35% larger than that achieved in the naturally cooled Astaloy CrM steel with the same carbon content.

As shown in Figure 5, a mono-phase of martensite was formed in the Astaloy CrM steel after sintering at 1120 and 1250 °C with subsequently fast cooling. A positive effect of sintering temperature on fatigue strength is shown in Figure 1. In this case, the porosity of Astaloy CrM steel was considered the primary factor affecting the fatigue strength. The other reasons for superior fatigue strength of Astaloy CrM were that sintering necks were strengthened via reducing oxides on particle surfaces with rising sintering temperature [23], and microstructure was strengthened by the reducing fraction of cementite due to more carbothermal reduction at higher temperature [24].

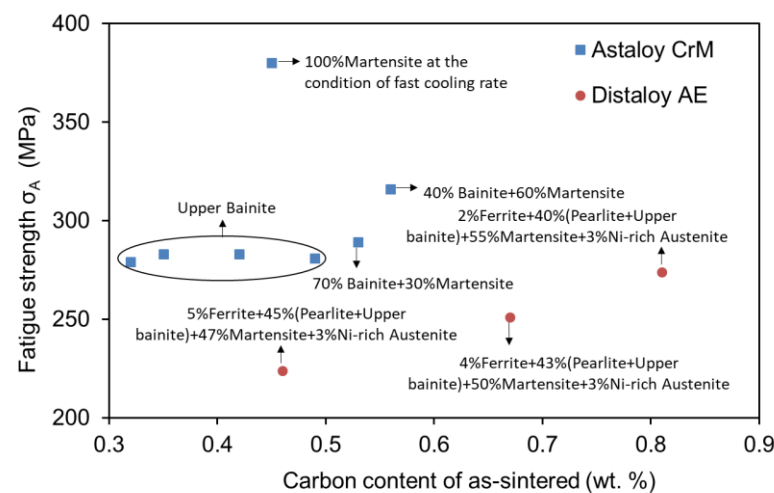


Figure 4. The variation of fatigue strength (σ_A) with the composition of complex phases with different carbon contents.

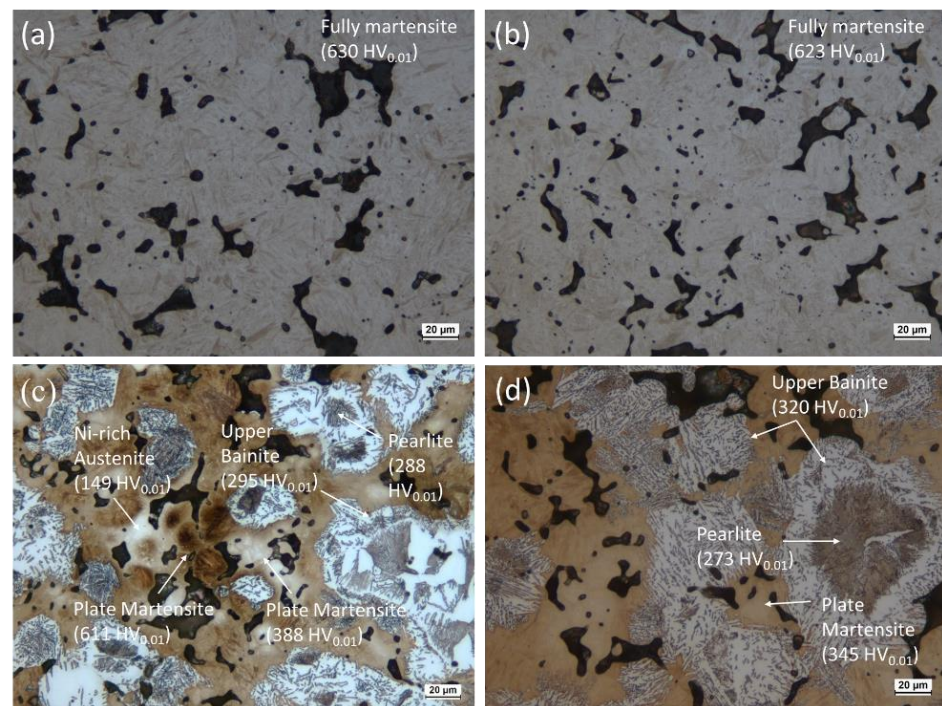


Figure 5. Porosity and phase of Astaloy CrM steel sintered at (a) 1120 °C and (b) 1250 °C and Distaloy AE alloy sintered at (c) 1120 °C and (d) 1250 °C.

As also indicated in Figures 4 and 5, a complex composition of phases, i.e., pearlitic, ferritic, bainitic, nickel-enriched austenitic, and martensitic, was simultaneously existed in the Distaloy AE steel sintered at 1120 °C and 1250 °C, respectively. A network surrounding the base powders was constructed by two types of phases, i.e., martensite lath, and lower bainite enriched with alloying elements of copper and nickel which caused solid-solution hardening by the enrichment of these elements in the vicinity of pores. A smaller fraction of the soft ferrite phase in ferritic and/or pearlitic-base powders, and accordingly larger fraction of high-strength plate martensite enriched with copper and nickel, were achieved by increasing carbon content in the Distaloy AE steel (with carbon content varying from 0.46 to 0.81 wt.%), which was beneficial for improving fatigue resistance. By increasing the sintering temperature from 1120 °C to 1250 °C, the distribution of alloying elements nickel, copper, and molybdenum could become more homogeneous, which reduced the fraction of

nickel-enriched austenite phase existing around pores. The same result was reported where heterogeneity was decreased at a higher sintering temperature in diffusion-bonding alloyed powder [25]. The area of phases of ferrite and/or pearlite was augmented by sintering at a high temperature of 1250 °C. However, an effect of softening in the sintering necks area was demonstrated by micro-hardness testing because more diffusion of alloying elements occurred from the surface to the interior of the powder matrix. As a negative result, fatigue strength was impaired when cracks start to nucleate at porosity. Cyclic plasticity and fatigue resistance were considerably damaged by decreasing the heterogeneity of phases with an attribute of being highly sensitive to cracks [26]. Obviously, the existence of the complex composition of phases due to the gradient of the compositions and its softening effect explains the non-significant effect of increasing sintering temperature on the fatigue strength in diffusion-bonding alloyed steel (Distaloy AE).

3.3. Fracture Behavior of Fatigue

The fracture behavior of fatigue cracks was inspected by optical microscopy. As shown in Figure 6a,c, cracks originated from pores close to the surface of the Astaloy CrM sample. This is consistent with the results reported elsewhere that fatigue failure is the result of the growth and coalescence of numerous surface (micro) cracks [27,28]. The propagation of the cracks occurred in sintering necks. For the Distaloy AE alloy steel, as shown in Figure 6b,d, the cracks initiated from pore clusters on the surface. The propagation of cracks was preferred to occur in the interior of the base powder composed of soft ferrite/pearlite with a lower yield threshold, consistent with the results reported in the study [29].

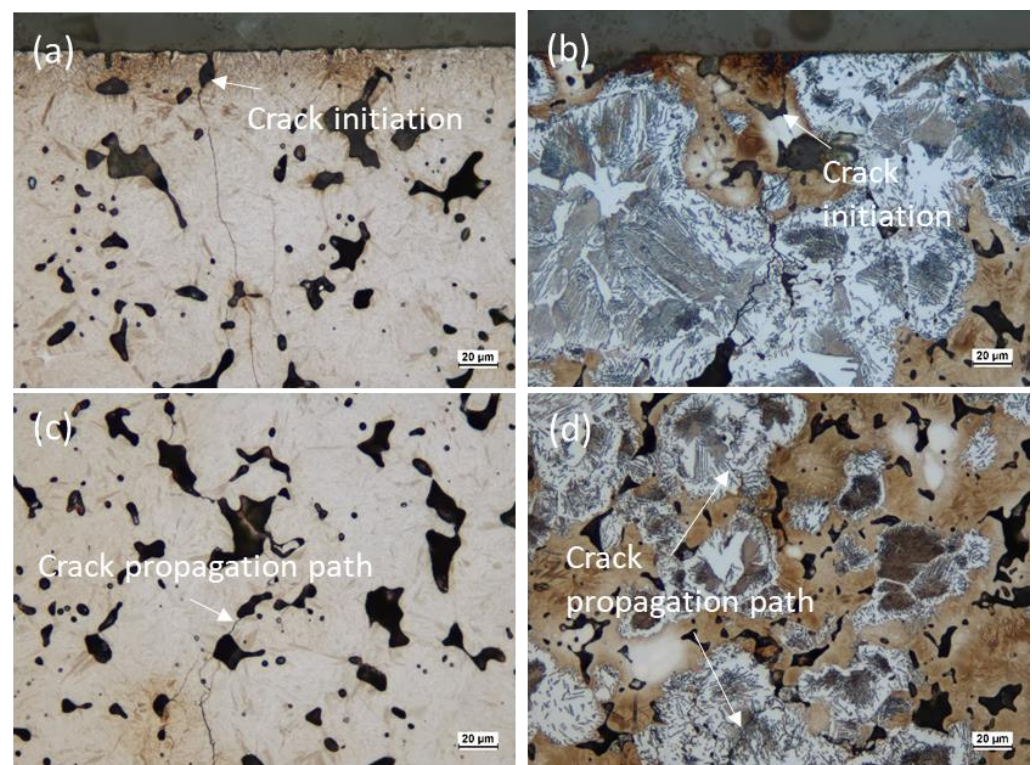


Figure 6. The origination and propagation of fatigue cracks: (a) crack initiation and (c) crack propagation path in Astaloy CrM, (b) crack initiation, and (d) crack propagation in Distaloy AE.

As shown in Figure 7 the path of the crack maintained a nearly straighter perpendicular to the edge at a 1250 °C sintering temperature in comparison with 1120 °C. The carbides in bainite/pearlite can arrest and hinder crack propagation, thus the cracks tend to be deflected when propagating in pearlitic regions [10,30]. With a decrease in pearlite fraction, the total propagation path of crack becomes flatter and straighter at the higher sintering temperature, which is unfavorable to fatigue property.

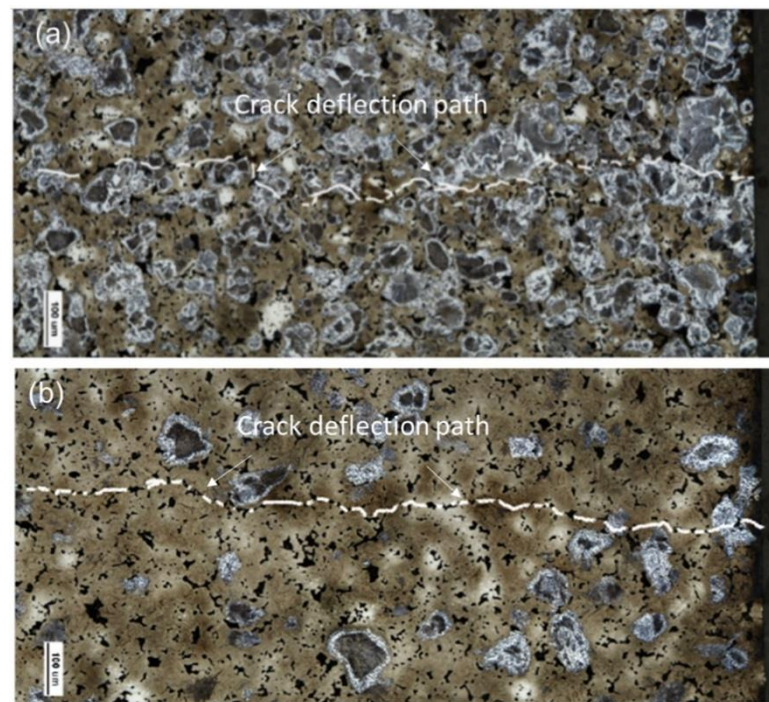


Figure 7. The propagation of fatigue cracks in Distaloy AE alloy steel sintered at (a) 1120 °C and (b) 1250 °C.

Figure 8 shows SEM fractography revealing fatigue fracture behaviors of diffusion-bonding alloyed steel (Distaloy AE) and pre-alloyed steel (Astaloy CrM), respectively. The fatigue fracture of the pre-alloyed Astaloy CrM steel is characterized by inter-particle fracture through a channel constructed by sintering necks. In other words, the origination of cracks occurred at sintering necks, and the propagation of them through the network of sintering necks resulted in an ultimate failure observed at the border between powders. This inter-particle fracture is attributed to the homogeneous distribution of a single phase from the border to the interior of powders, i.e., a fully developed mono-phase of martensite in the sintered Astaloy CrM steel. In contrast, the fatigue fracture mode of diffusion-bonding alloyed steel (Distaloy AE) is a trans-particle fracture with a big cleavage fracture through the interior of base powders due to a heterogeneous distribution of complex phases. Therefore, sintering necks were reinforced by the existence of a network of martensitic phases continuously formed along the borders of powders. Namely, the sintering necks possessed higher resistance to failure. As a result, the origination and propagation of cracks were preferred to take place in the interior of base powders composed of soft ferrite, pearlite, or bainite phase with lower yield thresholds.

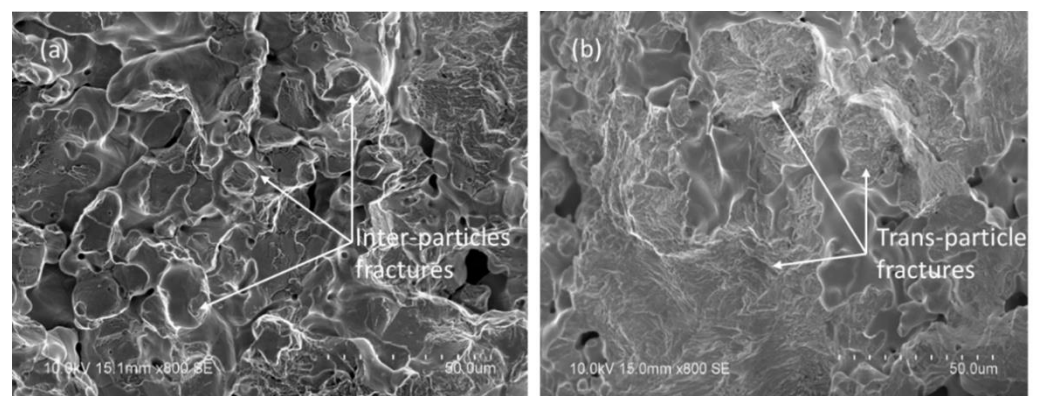


Figure 8. Fatigue fracture surfaces of powder metallurgy alloy steels: (a) Astaloy CrM exhibiting

inter-particle fracture through the channel constructed by sintering necks, and (b) Distalloy AE with trans-particle fracture occurred in the interior of base powders.

3.4. Quantitative Analysis of the Effects of Porosity and Phase Constitution on Fatigue Strength

The Murakami model is effective to predict the fatigue endurance limit and partition the contributions of the effects of microstructures and defects, from the geometric point of view, on the fatigue strength of steels manufactured by ingot metallurgy [31]. This model is adapted for materials with homogeneous microstructures, but not for heterogeneous structures [32]. In this study, a modified Murakami model was used to simulate the fatigue endurance limit of the powder metallurgy alloy steel with heterogeneous microstructure and composed of complex phases constitution. The modified Murakami model is given by

$$\sigma_M = \left[C \times (H_V + 120) / \left(\sqrt{RA} \right)^{1/6} \right] (E_p / E_0) \quad (1)$$

where, σ_M is the fatigue strength predicted by the formula, C is a constant, H_V is the Vickers micro-hardness tested in the area around defects, A is the total area of defects and soft phases in diffusion-bonding alloyed powders, R is the roundness of pores, E_p and E_0 are Young's modulus of porous and fully dense materials, respectively. Fatigue crack is mainly governed by localized yielding in the region around the crack tip under highly cyclic loading [33]. A separation of the influences of microstructure and porosity should be undertaken since the two factors can be independently considered. The Vickers micro-hardness is suitable to distinguish between metallographic phases and porosity. On the other hand, a defect area of initial crack growth is contained in a small volume where a concept called "initiation volume" was considered as A , which is highly involved in the variation of Young's modulus [7]. Thus, Young's modulus of porous and fully dense materials should be considered as the contribution of porosity to fatigue strength. Currently, the density of porous materials and Young's modulus have been established based on experience, while the MacAdam model directly gives the relationship between density and Young's modulus [34]. Fractography of cyclically loaded fracture samples revealed that cracks were preferably originated at large pores, indicating the constant C is 1.41 as edge defects [7], while the size and geometry of pores were considered as main factors in the new formula. Another point that must be further explored is the inferior performance of the diffusion-bonding alloyed steel compared to the pre-alloyed steel. This difference in fatigue performance is due to the presence of the nickel-enriched austenite phase in the diffusion-bonding alloyed steel. The austenitic is a soft phase where fatigue cracks can be easily nucleated. In this case, an interaction between pores and softer phases must be considered to estimate defect areas for the actual situation of crack propagation.

The calculated results by the modified Murakami model are shown in Figure 9. The fatigue strength estimated by the modified model is in good agreement with experimental results. Some deviation was observed for the cases at higher density and sintering temperature as it is difficult to properly measure the micro-hardness around pores and the sizes of pores, and the strength of sintering necks was ignored during calculation.

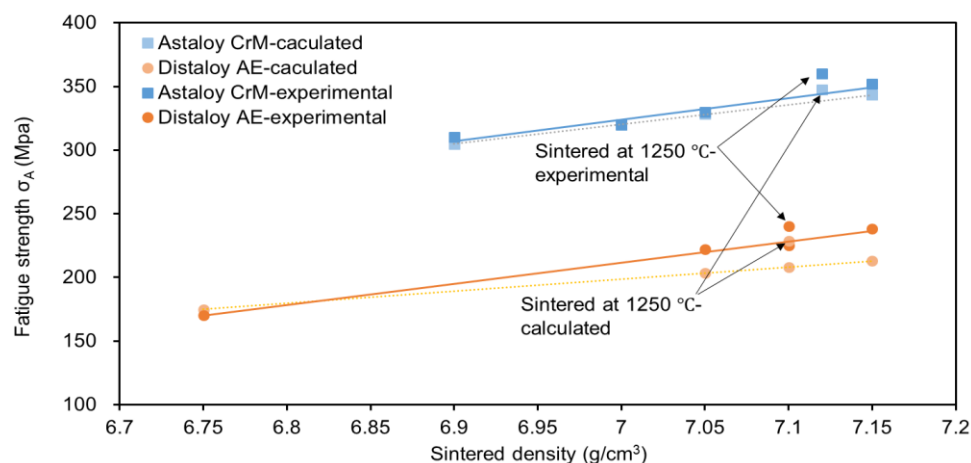


Figure 9. Calculated fatigue strength by the modified Murakami model as a comparison with the experimental results of fatigue tests.

4. Conclusions

Porosity and complex phases are crucial factors to the fatigue performance of alloy steels consolidated by powder metallurgy from pre-alloyed and diffusion-bonding alloyed powders. The pre-alloyed Fe-Cr-Mo steel (Astaloy CrM) is characterized by the homogeneous distribution of a mono-phase of martensite. However, heterogeneous distribution of complex phases and constitution of various types, e.g., ferritic, bainitic, nickel-enriched austenitic, and martensitic, was presented in the microstructure of the diffusion-bonding alloyed Fe-Cu-Mo-Ni steel (Distaloy AE). As a result, different modes of fatigue fracture were derived. Sintering necks were effectively reinforced in the Distaloy AE steel by the aid of a tough network consisting of the martensitic phase continuously distributed at sintering necks between powders. Therefore, the cracks initiate from the big pores on the surface and propagate preferably in the interior of the base powder matrix with a microstructure composed of soft phases (ferrite, pearlite, or bainite), resulting in a fracture mode of trans-particle fracture. In contrast, in the pre-alloyed Astaloy CrM steel with a microstructure consisting of a homogeneous mono-phase of martensite, micro-cracks were most likely to nucleate and then propagate through the channel constructed by sintering necks. In this case, an inter-particle fracture mode was observed at borders between powders. Moreover, for the Distaloy AE steel in which a tough network was constructed by martensite at sintering necks (the borders between powders), the fatigue strength was not significantly improved with increased sintering temperature. In this situation, the fatigue cracks are not easy to be initiated at the sintering necks. A modified Murakami model considering micro-scale defects and micro-hardness is effective to predict the fatigue performance of the alloy steels sintered from pre-alloyed and diffusion-bonding alloyed powders, respectively.

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