Improvement of mechanical strength of sintered Mo alloyed steel by optimization of sintering and cold-forging processes with densification

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Abstract. Powder metallurgy (P/M) materials have been expected to be spread in automotive industry. Generally, since sintered materials using P/M ones contain many pores and voids, mechanical properties of them are inferior to those of conventional wrought materials. To improve mechanical properties of the sintered materials, densification is effective. The aim of this study is to improve mechanical strength of sintered Mo-alloyed steel by optimizing conditions in sintering and cold-forging processes. Mo-alloyed steel powder was compacted. Then, pre-sintering (PS) using a vacuum sintering furnace was conducted. Subsequently, cold-forging (CF) by a backward extrusion method was conducted to the pre-sintered specimen. Moreover, the cold-forged specimen was heat treated by carburizing, tempering and quenching (CQT). Afterwards, mechanical properties were investigated. As a result, it was found that the density of the PS specimen is required to be more than 7.4 Mg/m³ to strengthen the specimen by heat treatment after CF. Furthermore, density and the microstructure of the PS specimen are most important factors to make the high density and strength material by CF. At the CF load of 1200 kN, the maximum density ratio reached approximately 99% by the use of the PS specimen with proper density and microstructure. At the CF load of 900 kN, although density ratio was high like more than 97.8%, transverse rupture strength decreased sharply. Since densification caused high shear stress and stress concentration in the surface layer, micro-cracks occurred by the damages of inter-particle sintered connection of the surface layer. On the contrary, in case of the CF load of 1200 kN, ultra-densification of the surface layer occurred by a sufficient plastic flow. Such sufficient compressed specimens regenerated the sintered connections by high temperature heat treatment and thus the high strength densified material was obtained. These processes can be applicable to near net shape manufacturing without surface machining.

1. Introduction

In recent years, automotive industry has promoted not only cost reduction but also solution of global environment problems. Those are enabled by saving resources and energy. To save them, strengthening materials and reducing used materials are effective. Sintered materials of iron series are able to be obtained with comparative ease. They can perform mass-production of small complicated...
shape parts, save resources and do not have to throw away garbage parts nearly. Furthermore, they enable the abbreviation of secondary machining in near net shape manufacturing and cost saving. For many years, sintered materials have not been used much in Japan as compared with Europe and USA. However, the use of sintered materials has increased year by year in Japan. Low-alloy steel powder including Cr, Ni, Mo, Cu and so on have been developed for high-strengthening of sintered materials of iron series [1-2]. Lowering mass of such added elements is expected for low costs. Since sintered materials contain many pores and voids within itself, they have a limit of strengthening. Especially, fatigue strength and wear resistance are decreased by stress concentration in pores of the surface layer. As has often been pointed out, one of effective strengthening processes is reduction of pores i.e. high densification. For this reason, sintering industry has used high temperature sintering and compression molding by high pressure. However, the general processes such as forging, sizing press and coining press cause small dimensional changes and can not strengthen the materials. As the post-compacting processes for densification, warm forming with a warming die and powder materials [3] and sinter-hot forging at high speed and high temperature have been also used [4-5]. These processes need a large scale facilities and high costs. Thus they cause the problems of lubricants shortage, die scoring and adhering oxidized scale. These problems spoil above advantages of sintering and its high cost performance. Conventionally, although wrought steel by machining and forging has been used for high strength automotive parts, such materials need high costs. Sintering and cold-forging (CF) have been used to manufacture densified powder metallurgy materials with near net shape [6]. These processes can produce the good exterior products and deform the materials at a low load to produce near net shape parts. Also, compared to other steel powder with many expensive elements, low cost iron powder is useful. Densification of those improves the mechanical properties (static strength, fracture toughness, fatigue strength, wear resistance and so on) of the powder material. However, there are few reports about sintering and cold-forging process for densified sintered materials [6]. Usually, the mechanical properties and macro deformation behaviours of the material have been investigated using the specimen that is cut out from the core area of it. The research on the surface layer of the near net shape sintered material with high density is little. In this study, the densified sintered material was produced with Mo diffusion alloyed atomized iron by pre-sintering and cold-forging. In particular, the optimum conditions of sintering and cold-forging were investigated to obtain superior mechanical properties by the use of the near net shape specimen with the natural forged surface.

2. Experimental procedure

2.1. Specimens preparation

In this study, Mo-alloyed steel powder (JFE steel, JIP SGM10MO-CMX) for cold-forging was prepared. This is atomized iron powder with diffusionally adhered 1 mass% Mo and additional 0.35 mass% C (graphite) to the surfaces of powder particles. The chemical composition of this iron powder and the particle distribution investigated by the maker are shown in table 1 and table 2, respectively. Figure 1 shows schematic of the fabrication process of the specimen. In this study, green compacting, pre-sintering (PS), cold-forging (CF) and heat treatment (CQT: gas carburizing, quenching and tempering) were conducted sequentially. Firstly, powder was compacted under pressure of 380-980 MPa using a floating die method. Density (ρc) of green compacts were approximately 6.8 Mg/m$^3$, 7.2 Mg/m$^3$ and 7.4 Mg/m$^3$ at pressure of 380 MPa, 620 MPa and 980 MPa, respectively. The width and the length of the

|  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|
| Element | C | Si | Mn | P | S | Mo | O | Additional graphite |
|---|---|---|---|---|---|---|---|---|
|---|---|---|---|---|---|---|---|---|
|  | 0.02 | 0.01 | 0.15 | 0.09 | 0.006 | 0.99 | 0.115 | 0.35 |

Table 1. Chemical composition of iron powder [mass%] (Lubricant: 0.5 mass%)
compact specimen were 10 mm and 54 mm, respectively. The height of them were 13, 12.5 and 12 mm for $\rho_c$ of 6.8, 7.2 and 7.4 Mg/m$^3$, respectively. PS was conducted by the use of a vacuum furnace at sintering temperature from 775°C to 1125°C and sintering time of 20, 50 and 80 min. The degree of vacuum was approximately 30 Pa. Then PS density ($\rho_{PS}$) was almost equal to $\rho_c$. Generally, decarburization in steel powder occurs and it causes to decrease the strength of the material after pre-sintering. Moreover, in case of the N$_2$-H$_2$ conveyer furnace for mass-production, surface decarburization occurs by discharging CO$_2$ derived from H$_2$O in the furnace. When the graphite on the surface of the particle is exposed to high temperature, it easily diffuses into the material and reacts with dissolved oxygen in the material. Thus, decarburization occurs and the strength of the pre-sintered material decreases. Usually, the green products are sunk in a tray filled with carbon powder and are sintered so that those are carburized little. However, it is not proper for sinter-forging because hardening and reducing of the deformability occur at PS. Therefore, the vacuum furnace should be used to avoid the above-mentioned problems. After PS, CF was conducted using backward extrusion with an oil hydraulic press machine (Tanakakame, TMS-200X100). The setting load range was from 400 kN to 1300 kN. Pressing time was 1 sec. After CF, heat treatment was conducted by the use of a vacuum carburizing furnace (Nakamihonrokgoyo, NVF-15). Gas carburizing was conducted at hold temperature of 900°C for 180 min. Quenching was conducted by oil quenching (quenching conditions: 840°C, 60 min). Tempering was conducted at 180°C for 90 min. Before heat treatment, the flash formed by cold-forging was cut as shown in figure 1.

2.2. Experiment and analysis

Dimensions, density ratios and microstructures of specimens were investigated after each process. Density was measured as apparent density by the Archimedes method. The Charpy impact test was conducted using non-notched specimens with a 500 kJ Charpy tester and an instrumented 300 kJ Charpy tester (Tokyo testing machine, IC-50 and CIEM-300D). Transverse rupture strength was measured using a fatigue testing machine (Shimadzu, EHF-EM100kN/1kNm). Fracture surfaces were observed using a field-emission scanning electron microscope (FE-SEM) after tests. Hardness measurement was conducted with a rockwell hardness tester and a micro vickers hardness tester. Microstructural observation was conducted with an optical microscope and a SEM. Mapping analysis and quantitative analysis were conducted using an electron probe X-ray microanalyzer (EPMA). Quantitative analysis of carbon in the pre-sintered specimen was conducted by combustion and infrared absorption spectroscopy. For analysis, test pieces of 0.5 g sliced from the core of the pre-sintered specimen was prepared. The cross section polisher by Ar-ion beam was used when it is difficult to observe a slight change of microstructures by conventional mechanical polishing.

| Table 2. Particle size distribution of iron powder |
|---------------|---------|---------|---------|---------|---------|---------|---------|
| Particle size [μm] | over 180 | 150 ≤ 180 | 106 ≤ 150 | 75 ≤ 106 | 63 ≤ 75 | 45 ≤ 63 | below 45 |
| Ratio [%] | 0.1 | 7.4 | 18.0 | 24.7 | 7.3 | 20.2 | 22.3 | 100 |

**Figure 1.** Schematic of fabrication process of specimen.
3. Results and Discussion

3.1. Properties of pre-sintered specimens

3.1.1 Impact properties and fracture surface. Figure 2 shows results of the Charpy impact test. In the figure, average Charpy impact energies of three times measurement are plotted. As has often been pointed out, the Charpy impact value increases exponentially with densification [7]. The similar trend was observed in this study. With higher density of the green compact and higher PS temperature, Charpy impact energy is higher too. In the upper optical images in the figure, two typical fracture surfaces are shown. Right side and left side images show the fracture surfaces of specimens with high and low impact energy, respectively. In case of high impact energy, light silver grains were observed in the fracture surface. It corresponds to cleavage fracture confirmed by SEM. Exactly, cleavage fracture area is caused by hardening with advanced sintering and transgranular fracture. On the other hand, grain boundary fracture occurs due to insufficient sintering in the specimen with low impact energy and the frosted gray area was observed as an uneven fracture area with many pores.

3.1.2 Morphology of side surfaces of specimens. Figure 3 shows back-scattered electron images of the side surfaces of pre-sintered specimens at various PS temperatures. Since the side surface of the specimen did not contact with the upper punch directly, superficial depression of boundaries occurred by growth of sintering connection on the side surface. To increase toughness and density of the specimen by CF, proper morphology of the side surface of the specimen after PS is required. From figure 3, the sufficient sintering surface was confirmed in the image at PS temperature above 975°C because PS temperature is approximately over A3 transformation point of steel used. On the basis of these results shown in figure 2 and 3, we selected 7.4 Mg/m³ as proper density of green compact and 1000 - 1100°C as proper PS temperature, respectively.

3.1.3 Hardness and microstructure. Table 3 shows contents of carbon and hardness of core areas of pre-sintered specimens. When PS temperature increased, although the carbon content in the core area of the specimen decreased, the Rockwell hardness (HRA) increased. HRA saturated at PS temperature above 1050°C. This means that

![Figure 2. Effect of density of green compact and PS temperature on Charpy impact energy (PS time: 20 min).](image-url)
sufficient sintering was conducted at those temperatures and that hardening by sintering exceeded the weakening by decarburization. Figure 4 shows microstructures of core sections of pre-sintered specimens. White phases and gray phases in the figures are ferrite and pearlite, respectively. It seems that the pearlite area increases with increasing PS temperature. Also, as shown in table 1, the powder material contains 0.115 mass% O and a little lubricant. As shown in tables 1 and 3, in case of vacuum sintering, it was found that 0.04-0.07 mass% graphite is decarburized by oxidizing of graphite contained in the material, instead of oxidizing carbon of lubricant. Therefore, it was clarified that the weight of added carbon is desirable to be a half of the content of dissolved O in addition to the weight of original carbon.

3.1.4 Transverse rupture strength (TRS). Figure 5 shows TRS of pre-sintered specimens under various PS conditions. TRS is relatively stable at PS temperature above 1050°C, irrespective of sintering time. Generally, the possible maximum temperature in mass production is approximately 1100°C. Therefore, recommended PS temperature and time seem to be 1050°C-1075°C and 20 min, respectively. The pre-sintered material under such conditions has both high strength by pearlite and deformability by ferrite, and it is economical. The previous studies reported that Mo promotes sintering [8] but Mo alloying in ferrite is few [9]. Similar tendency is confirmed in the hardness shown in table 3.

| PS temperature | C [mass%] | H_{RA} | HV0.2kgf (Pearlite) | HV0.2kgf (Ferrite) |
|----------------|-----------|--------|---------------------|---------------------|
| 975°C          | 0.31      | 44.4   | 201                 | 130                 |
| 1000°C         | 0.30      | 45.0   | 198                 | 128                 |
| 1025°C         | 0.30      | 46.0   | 199                 | 123                 |
| 1050°C         | 0.29      | 47.3   | 197                 | 129                 |
| 1075°C         | 0.28      | 47.6   | 197                 | 123                 |
| 1100°C         | 0.28      | 47.4   | 205                 | 134                 |

Figure 3. Back-scattered electron images of side surfaces of specimens after PS (\(\rho_c\): 7.4 Mg/m³, PS time: 20 min).

Figure 4. Microstructures of core sections of PS specimens (\(\rho_{PS}\): 7.4 Mg/m³, PS time: 20 min).

Table 3. Results of quantitative analysis of carbon and average hardness for core area of PS specimen (\(\rho_{PS}\): 7.4 Mg/m³, PS time: 20 min).
Figure 5. Effect of PS temperature and time in transverse rupture strength ($\rho_{PS}$: 7.4 Mg/m$^3$).

![Effect of PS temperature and time in transverse rupture strength](image)

Figure 6. Average density of cold-forged specimens ($\rho_{PS}$: 6.8, 7.2, 7.4 Mg/m$^3$).

(a) Average density at CF load range of 400-1300 kN (b) Magnified graph of (a)

3.2 Properties of cold-forged specimens

3.2.1 Effect of PS conditions on cold-forged (CF) specimens.

Figure 6(a) and 6(b) show densification properties of PS specimens by CF. Density ($\rho_{CF}$) of the specimen saturated at the setting CF load above 1100 kN. At such CF load, with increasing initial PS density ($\rho_{PS}$), an upper limit of $\rho_{CF}$ increases. This modification of density ($\rho_{CF}$) scarcely depend on PS temperature.

Also, maximum CF density ($\rho_{CF}$) is from 7.78 to 7.81 Mg/m$^3$ for each PS condition. These density are higher than that of the previous study by using free cold upsetting [10]. Thus, the proper density of the specimen after PS ($\rho_{PS}$) seems to be 7.4 Mg/m$^3$. Figure 7 shows microstructures of cross sections of cold-forged specimens at various CF loads. In case of setting CF load of 1100 kN, it was found that ultra-densification occurs on the surface layer by the plastic flow of ferrite phases. The similar densified microstructures were observed in cold-forged specimens at the setting CF load above 1100 kN, and the number of pores decreased with increasing the CF load. Since the CF load of 1100 kN with the upper limit of $\rho_{CF}$ in figure 6 equals to the load for the flat ferrite grains elongation in figure 7, these microstructural deformations of the surface layer will be related to average density of the CF specimen ($\rho_{CF}$).
Figure 7. Microstructures of cross sections of cold-forged specimens at various CF loads ($\rho_{PS}: 7.4 \text{ Mg/m}^3$, PS conditions: 1050°C, 20 min).

| Optical microscope images | Setting CF Load |
|--------------------------|----------------|
| ![Microscope Images](image1) | 600 kN |
| ![Microscope Images](image2) | 900 kN |
| ![Microscope Images](image3) | 1100 kN |

(a) Flash height and average density of cold-forged specimen ($\rho_{CF}$) in case of PS temperature of 1075°C.

(b) Flash height and average density of cold-forged specimen ($\rho_{CF}$) in case of $\rho_{PS}$ of 7.4 Mg/m$^3$.

Figure 8. Relationship between flash height and average density after cold-forging.
3.2.2 Flash height and average density. Figure 8 shows a relationship between the flash height and average density (ρ_{CF}). From figure 6(a) and 8, it was found that the flash increases exponentially with the higher CF load. Moreover, at the CF load range investigated in this study, in case of a low initial PS density (ρ_{PS}), final flash heights were higher. Regardless of ρ_{PS}, ρ_{CF} approached to almost the same value. Also, as shown in figure 8(b), in case of the same initial PS density (ρ_{PS}), the effect of PS temperature of the flash height is negligible. This result means that the initial PS density (ρ_{PS}) is an important factor to obtain higher average density (ρ_{CF}).

3.3. TRS and microstructure after carburized quenching and tempering (CQT)

Figure 9 shows TRS of PS-CF specimens after CQT heat treatment. In case of the range of the CF load is from 400 kN to 800 kN, TRS gradually increases with increasing the CF load. As the previous studies indicated, generally, TRS is directly proportional to a sintered density [11]. However, TRS decreases sharply at the CF load range from 800 kN to 900 kN. With increasing the CF load above 900 kN, TRS also increases. Such change in TRS with the change of the CF load is a “death valley” type. Similar phenomena were reported as “abnormal brittleness” in results of decreasing Charpy impact values at using specimens with the densities of 7.2-7.4 Mg/m^3 [12-14]. However, results of this study in higher density range could indicate a different trend of the transverse rupture strength. At the CF load above 900 kN, irrespective of PS temperature, TRS increased gradually again. In case of the CF density (ρ_{CF}) of 7.70-7.76 Mg/m^3 (density ratio: 97.8-98.5%), TRS decreases and not suitable for production of near net shape parts.

Figure 10 shows back-scattered electron images of cross sections of surface layers after cold-forging. Figure 10 (a) and (b) show the residual pores existed on the surface layer. In figure 10 (c) and (d), channelling contrasts indicating a large scale of a plastic flow are observed. In case of image (c) at the CF load of 950 kN, although pores were compressed, micro-cracks formed at triple or quadruple points of grain boundaries in the surface layer. Such micro-cracks cause TRS decreasing. When a high pressure is loaded on the surface layer for ultra-densification, inter-particle sintered connection is damaged. Then, the micro-cracks along grain boundaries are introduced by stress concentration and shearing stress derived from a compression stress and a plastic flow.

As shown in figure 6(a) and figure 7, the CF load above 1100 kN is needed to achieve the maximum density. Furthermore, for ultra-densification of the surface layer and disappearance the
micro-cracks, a sufficient plastic flow is required as shown in figure 10(d). The setting CF load of 1200 kN can compress the specimen sufficiently and heat treatment at high temperature could regenerate the sintered connection. These processes could give high strength densified materials stably and be applicable to not only the improvement of spalling fracture on the surface of densified sintered iron [15] but also the near net shape manufacturing by sinter forging.

Figure 10. Back-scattered electron images of cross sections of surface layers after cold-forging 
(ρPS: 7.4 Mg/m³, PS temperature:1050°C, PS time: 20 min).

4. Conclusions
In this study, we made the high density sintered material with Mo diffusion alloyed atomized iron by sintering and cold-forging. Then the optimum conditions of pre-sintering (PS) and cold-forging (CF) to obtain the good mechanical properties were investigated. The obtained results are as follows.

- Since the specimen is decarburized a little in vacuum sintering, the weight of added carbon is desirable to be a half of the content of dissolved O in addition to the weight of original carbon.

- Density of PS specimen is necessary more than 7.4 Mg/m³ to obtain good mechanical properties. Furthermore, in case of PS process time of 20 minutes, PS temperature is necessary more than 1050°C. The microstructure and density of the PS specimen are most important for high densification by CF. The maximum density became approximately 7.8 Mg/m³ (density ratio: 99%) at the setting CF load of 1200 kN in case of the backward extrusion.
• At the setting CF load range from 800 kN to 900 kN, TRS decreased sharply. The safety threshold level of the density ratio was approximately under 97.8% (7.70 Mg/m$^3$) or over 98.8% (7.78 Mg/m$^3$) to obtain high TRS. When the densification of the core of the sintered specimen progresses, shearing stress generates in the surface layer and stress concentration occurs in the pores of the surface layer. Thus inter-particle sintered connections are damaged and micro-cracks initiate in the surface layer. Moreover, in case of the backward extrusion, ultra-densification of the surface layer occurs by the sufficient plastic flow at the setting CF load of 1200 kN. Such sufficient compressed specimens could regenerate the sintered connections by heat treatment at high temperature and could obtain the high strength densified materials stably.

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