学 位 論 文 の 要 旨

Mo 系焼結鋼の冷間鍛造および熱処理による機械的特性向上に関する研究

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本研究では，Mo 系部分拡散合金鋼粉を用い，その焼結冷間鍛造材の機械的特性，特に疲労強度を向上させるための各工程における有効条件を明らかにすることを目的とした．圧粉体成形，一次焼結，冷間鍛造，最終熱処理の各工程の組合せにより，最終的に機械的特性に優れる焼結冷間鍛造熱処理材を得ることに成功した．また，製品設計で重要となる大塑性変形を伴う鍛造による形状および密度変化の推定を可能とする有限要素(FEM)解析手法を提案した．

第 1 章では，粉末冶金の歴史から，一般機械部品や自動車部品における焼結材の位置付けと生産動向，鉄基焼結材およびその高強度化手法，高強度焼結部品の開発に向けた研究課題，本研究の目的および論文の構成について述べた．

第 2 章では，圧粉体成形から冷間鍛造までの工程が，金属組織，密度および塑性変形能に及ぼす影響に着目し，鍛造による高密度化特性および塑性変形能に優れた焼結冷間鍛造材を得るのに有効な工程条件を検討した．

一次焼結温度を上げると，フェライト系組織が減少しパーライト系組織が増加する傾向が確認された．これは，一次焼結温度の上昇に伴い，Fe 粉の表面に付着していたグラファイト(C)の Fe 粒内への拡散の進行に起因することを示した．一次焼結体密度と一次焼結時間が一定の場合，一次焼結温度に依存せず，鍛造後の予肉の高さは，鍛造荷重を増加させた際の密度の上昇に伴い指数関数的に上昇することを確認した．また，一次焼結体密度が高いほど，冷間鍛造による限界密度も高くなることを示した．限界密度に達するのに必要十分な設定鍛造荷重は，本研究で用いた角形焼結試験片では 1200 kN であり，その際の限界密度は 7.8 Mg/m^3 以上となることを示した．さらに，一次焼結温度および冷間鍛造後の密度が同じであっても，焼結時間 60 min では気孔の球状化が促進されるため，焼結時間 20 min に比べ，塑性変形能に優れることを示した．

これらの成果より，一次焼結条件 1075℃, 60 min で密度 7.4 Mg/m^3 の焼結体は，塑性変形能の低いパーライト系組織が増加するにもかかわらず，高密度化特性と塑性変形能に優れることを示した．

第 3 章では，機械的特性に及ぼす焼結，冷間鍛造および浸炭熱処理の各工程条件の影響

を明らかにし, 第 2 章の調査結果と併せて機械的特性の向上に有効な工程条件を検討した.
一次焼結条件や一次焼結体密度にかかわらず, 冷間鍛造により平均密度を約 7.7 Mg/m^3 とすると，表層域に極微細な粒界き裂が発生し，鍛造材の衝撃値の低下および浸炭熱処理後の曲げ強度の低下を招くことを明らかにした．一方, 冷間鍛造で平均密度を 7.8 Mg/m^3 以上に高密度化すると，微細き裂が圧接され，ガス浸炭熱処理による素材全体における体拡散と拡散接合を利用することによりき裂の消失が促進されることを明らかにした. その結果, 平均密度約 7.7 Mg/m^3 の時の曲げ強度から強度が回復することを示した．さらに，この熱処理材の強度回復効果が高くなる有効条件を明らかにした．その条件として，一次焼結体密度が 7.4 Mg/m^3 で，一次焼結条件は，ニアネットシェイプに適した 1050℃，20 min，および塑性変形能に優れる 1075℃，60 min が望ましく，設定冷間鍛造荷重 1200 kN で密度 7.8 Mg/m^3 以上とすることが望ましいことを示した.
第 4 章では，第 3 章で得られた有効な一次焼結条件，冷間鍛造条件を用い，これに新たな浸炭熱処理法を適用することによる素材の高疲労強度化を試みた．ガス浸炭熱処理よりも更なる高温処理が可能な真空浸炭熱処理による金属組織の改質を図り，各種熱処理条件が金属組織および疲労強度に及ぼす影響を調査した. 鍛造による高密度化で Fe 粒子間の接触面積が増加すると, 熱処理時に極低炭素の Mo リッチ部で γ-Fe が α-Fe に変化し，α-Fe 特有の高速な体拡散が支配的となり, Fe の自己拡散が著しく促進されることを明らかにした. 高密度焼結冷間鍛造材に対し 1000℃，60 min の二次焼結を施すと，素材心部でも C，Mo および Fe の拡散が進み，鍛造で表層に生成された極微細な粒界き裂が拡散接合により消失し，残留気孔が球状化する．そのような材料では，優れたシャルピー衝撃値(437 J/cm^2)が得られることを示した．さらに，高濃度炭素下での真空浸炭浸窒熱処理を提案した．本手法では，耐摩耗性に優れる窒素マルテンサイトと球状セメンタイトを含む硬い表面および深い硬化層を生成しつつ，表層に多量の残留オーステナイト組織を生成することにより，き裂進展を抑制することに成功した．その結果，高疲労強度の焼結材を実現した．
第 5 章では，焼結冷鍛材の形状および密度を予測することを目的とし，角形焼結試験片を後方押出し式で冷間鍛造する工程の三次元 FEM 解析を試みた. 焼結体の真応力－真ひずみ線図を取得して，実験値と計算値の応力比の多項式近似により，多孔質体の FEM 解析に有効な補正真応力－真ひずみ線図を求める手法を確立した．FEM 解析に，補正真応力－真ひずみ線図，リメッシュおよび要素消去法を用いることで，形状および密度について解析が可能となることを示した．さらに，冷間鍛造工程においてパンチに作用する荷重の解析も可能となることを示した.
第 6 章では，本研究を総括した.

学 位 論 文 の 要 旨

Study on improvement of mechanical properties of Mo system sintered steel by cold forging and heat treatment

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The purpose of this study is to obtain proper process conditions to improve mechanical properties of sintered and cold-forged materials using Mo-partial-diffusion alloyed steel powder, in particular, their fatigue properties. The sintered, cold-forged and heat-treated material that has superior mechanical properties was obtained successfully by optimizing green compacting, primary sintering (PS), cold forging (CF) and heat treatment together. Also, since the changes in the shape and the density of the sintered material by cold-forging with large plastic deformation is important in the product design, the new finite element method (FEM) that can estimate them were suggested.

In chapter 1, the history of powder metallurgy, positioning and production trends of sintered materials in mechanical and automobile parts, Fe-based sintered materials and their strengthening methods, the problems in research to develop high-strength sintered parts, were described. Moreover, the purpose of this study and the consist of the thesis were also described.

In chapter 2, the effects of processes from green compacting to cold forging on the microstructure, the density and deformation properties of the specimen were investigated. Effective process conditions were investigated to obtain the sintered and cold-forged material that has good densification properties and the deformability by cold-forging.

With increasing the PS temperature, the ferrite and the pearlite microstructures were decreased and increased, respectively. This is caused by promotion of the diffusion of carbon in the grain of Fe with increasing the PS temperature. When both of the density of the primary-sintered specimen and the PS time were constant, the height of flash after CF exponentially increased with the densification by increasing the CF load regardless of the PS temperature. Also, the limit density by CF became high with increasing the density of the primary-sintered specimen. In the square shape specimen used in this study, the set CF load that is sufficient to reach the limit density was 1200 kN. In that case, the limit density became more than 7.8 Mg/m^3. Moreover, it was confirmed that the specimen sintered for 60 min has the superior deformability compared with that sintered for 20 min due to the promotion of spheroidizing pores even if the PS temperature and the density of cold-forged specimen are constant.

From these results, it was found that the specimen, which is sintered at 1075°C for 60 min and has the density of 7.4 Mg/m^3, has superior densification properties and deformability although the perlite microstructures with low deformability are increased.

In chapter 3, the effect of process conditions in PS, CF and carburizing heat treatment on mechanical properties of the specimen was investigated. Considering the results obtained in chapter 2, proper process conditions were examined to improve the mechanical properties.

Regardless of PS conditions and the density of the primary-sintered specimen, when the average density becomes approximately 7.7 Mg/m^3 by CF, microcracks form in the surface layer of the specimen and thus both charpy impact strength after CF and the bending strength after carburizing heat treatment are reduced. On the contrary, in the specimen which average density more than 7.8 Mg/m^3 is densified by CF, microcracks are press-contacted and disappearance of the microcracks is promoted by the volume diffusion in the whole specimen and the diffusion bonding in gas-carburizing heat treatment. As a result, the bending strength of the specimen with 7.8 Mg/m^3 average density recovers compared to that with 7.7 Mg/m^3 average density. Moreover, effective process conditions to such strength recovery effect for the heat-treated specimen were revealed. As the conditions, it was shown that it is desirable that the density of primary-sintered specimen is 7.4 Mg/m^3 and the PS conditions are at 1050°C for 20 min and at 1075°C for 60 min in case of near-net shaping and large deforming, respectively. Moreover, it was shown that it was desirable the average density after CF becomes more than 7.8 Mg/m^3 using the set CF load of 1200 kN.

In chapter 4, new carburizing heat treatment method was developed to achieve the high fatigue strength of the specimen using effective PS-CF conditions obtained in chapter 3. Using vacuum carburizing heat treatment that can conduct high temperature processing compared to gas-carburizing heat treatment, reforming of microstructure was performed and the effect of heat treatment conditions on the microstructure and fatigue strength of the specimen was investigated. When the contact area of Fe particles increases with densification by CF, γ-Fe transforms to α-Fe in the Mo-rich area with extra low carbon during heat treatment. Then, the high speed volume diffusion peculiar to α-Fe becomes dominant and thus the self-diffusion of Fe is promoted remarkably. When secondary sintering is conducted to the sintered and cold-forged specimen with high density at 1000°C for 60 min, the diffusion of C, Mo and Fe progresses even in the center area of the specimen. Then, the microcracks formed at the grain boundaries in the surface layer by CF are disappeared by diffusion bonding and residual pores are spheroidized. In such specimen, the superior charpy impact value (437 J/cm^2) was obtained. Moreover, in this study, vacuum carbonitriding heat treatment under highly-concentrated carbon was suggested. Using this method, the hard surface with a deep hardening layer, which includes both the nitrogen martensite and the spherodized cementite with superior wear resistance, can be formed. In addition, the excessive retained austenite can be formed in the surface layer. In the specimen with such microstructures, progress of cracks is suppressed. As a result, the sintered material with high fatigue strength was realized.

In chapter 5, 3D-FEM analysis that simulates the CF process of the backward extrude forging of the square shape specimen was performed to predict the shape and the density of the sintered and cold-forged material. At first, the true stress-true strain (S-S) diagram of the sintered material was obtained experimentally. The method to obtain the corrected S-S diagram for FEM analysis of the porous material was established using the polynominal approximation of stress ratios of experimental values and calculated values. It was shown that the shape and the density can be analyzed using the corrected S-S diagram, the remeshing method and the element elimination method. Moreover, it was also shown that the load to act on the punch in CF can be analyzed using the developed method.

In chapter 6, this study was summarized.