Strength evaluation of fine grained steel and microwave sintered ceramics using X-ray diffraction technique

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Abstract

This thesis is aimed to evaluate the strength characteristics of fine grained steel and microwave sintered ceramics, which have smaller grain size compared to conventional steel and ceramics under various processing conditions or environment by using the X-ray diffraction technique. In this thesis, X-ray residual stress measurement, as one of the most common techniques that is used to measure residual stresses is introduced in detail. Then, the behaviors of residual stress distribution caused by directionality mechanical processing of fine grained steel were investigated with the tri-axial stress analysis method and Debye ring observation. The stress corrosion resistance property of the fine grained steel was also evaluated via SCC (Stress Corrosion Cracking) test. After SCC test, the residual stress distributions in the fracture surface were investigated by X-ray diffraction technique. According to the experiment results, fine grained steel is shown to have better stress corrosion cracking resistance than conventional steel. On the aspect of microwave sintered ceramics, some advantages of microwave heating technology were confirmed via the comparison of the rate of volume change, bulk density, water absorption and apparent porosity of microwave and conventionally sintered samples. In addition, the homogeneous performance of the microwave sintered ceramics was evaluated on the microscopic scale by the thermal residual stress and the pore ratio investigations. It was confirmed that the sinterability of ceramics is homogenously improved by hybrid microwave sintering.

論文要旨

On production or manufacturing fields, the development of the products which possess high performance usually need high strength materials. Therefore, the importance of high strength materials have been paid attention to by the researchers more and more, and a variety of strengthening mechanisms were developed. Grain boundary strengthening is one of these mechanisms, this mechanism increase the strength via decreasing grain size of material. A important merit of grain boundary strengthening compared other strengthening mechanisms is that it not only improve the strength but also increase the toughness. In addition, grain boundary strengthening has still following advantages: 1) increasing strength without the changes of composition; 2) ecology conservation because of more recyclable to materials and rare metal saving; 3) more economical cost of materials because of energy and resource saving. In the present study, two types of special new materials: fine grained steel and microwave sintered ceramics, which have smaller grain size compared to conventional steel and ceramics were emploied to investigate and evaluate their strength behavior.

Fine grained steel and microwave sintered ceramics would be used on many fields as new generation's high strength materials. Therefore, it is necessary to realize their strength characteristic under various processing conditions or environment. With this aim, this thesis is to discuss the strength behaviors of fine grained steel and microwave sintered ceramics materials after appropriate processing respectively, via various experimental methods.

In this thesis, the behaviors of residual stress distribution caused by directionality mechanical processing of fine grained steel were investigated with the tri-axial stress analysis method and Debye ring observation. The stress corrosion resistance property of the fine grained steel was also evaluated via SCC (Stress Corrosion Cracking) test. On the aspect of microwave sintered ceramics, some advantages of microwave heating technology were confirmed via the comparison of some important parameters of microwave and conventionally sintered samples. In addition, the homogeneous performance of the microwave sintered ceramics was evaluated on the microscopic scale.

The thesis consists of 7 chapters, and the important contents and results of each chapter are summarized as follows:

Chapter 1 is the general introduction of this thesis. In this chapter, background, objectives and contents of the thesis are introduced briefly.

In Chapter 2, X-ray residual stress measurement, as one of the most common techniques that is used to measure residual stresses is introduced in detail.

The residual stress measurements using X-ray diffraction technique is considered as nondestructive method. This method basically measures the angles at which the maximum diffracted intensity take place when a crystalline sample is subjected to x-rays. From these angles, it is possible to obtain the interplanar spacing of the diffraction planes using Bragg's law, which can be expressed as $n\lambda = 2d\sin\theta$, and the interplanar spacing is used as a strain gage. If the residual stresses exist within the sample, then the residual stresses cause changes in the spacing of the lattice planes from their stress free value to a new value that corresponds to the magnitude of the residual stresses.

The fundamental equation that is used in x-ray diffraction strain measurement can be expressed as

$$(\varepsilon'_{33})_{\phi\psi} = \frac{d_{\phi\psi} - d_0}{d_0} = \varepsilon_{11}\cos^2\phi\sin^2\psi + \varepsilon_{12}\sin 2\phi\sin^2\psi + \varepsilon_{22}\sin^2\phi\sin^2\psi$$

$$+ \varepsilon_{33}\cos^2\psi + \varepsilon_{13}\cos\phi\sin 2\psi + \varepsilon_{23}\sin\phi\sin 2\psi \quad (7\text{-}1)$$

As a result of x-ray diffraction, there are three basic behaviors of $d_{\phi\psi}$ vs. $\sin^2\psi$ for a d-spacing at all ψ tilts, and they are linear behavior, ψ-splitting behavior and oscillatory behavior respectively. For both linear behavior and ψ-splitting behavior, equation 7-1 can be used to determine the strains from the given data, then calculating residual stresses value. However, the data of oscillatory behavior can not be solved with the equation 7-1.

In Chapter 3, for clarifying the influence on the residual stress distribution of the directionality processing method and the grain size, specimens of fine grained steel and conventional steel were processed by mechanical grinding and emery polishing respectively. Then the behaviors of residual stress distribution were investigated by X-ray diffraction technique with the tri-axial stress analysis method. At the same time, Debye ring observation was carried out, and the results were compared with those of tri-axial stress analysis.

The main results are summarized as follows:

(1) It was confirmed by the observation of IP images that the grain size of fine grained steel is bigger than that of conventional steel.
(2) In both NFG600 and SM490, the Tri-axial stress state is caused by the directionality processing.
(3) In the case of mechanical grinding, it was confirmed that the maximum depth of the processing influence of NFG600 is bigger than that of SM490.
(4) It was shown that the disappearance of σ_{13} and σ_{23} corresponded to the changes of the IP images. According to this result, it is possible to connect the first kind stress with the third kind stress by using IP images.
(5) It is considered that the maximum depth of the processing influence is bigger than the settling point of Tri-axial stress.

In Chapter 4, for a better understanding of the strength characteristic and environmental characteristic of fine grained steel under corrosion environment, SCC test was carried out. Subsequently, fracture surface was observed with SEM images and the residual stress distributions in the fracture surface were investigated by X-ray diffraction technique. The difference of stress corrosion resistance property between fine grain steel and conventional steel was evaluated.

The main results are summarized as follows:

(1) Fine grained steel NFG600 is shown to have better stress corrosion cracking resistance than SM490 as measured by SCC test.

(2) On both NFG600 fracture surface and SM490 fracture surface, the coexistence of fracture ductility and the form of embrittlement is formed. However, SM490 causes the fracture ductility more easily than NFG600. Therefore, it is possible that the crack size is related to the grain size of the material.
(3) The difference of plastic zone size ω_y due to the difference of the crystal grain size was confirmed. ω_y of NFG600 is smaller than ω_y of SM490 because fine grain steel NFG600 has stronger yield strength.

In Chapter 5, for confirming the advantages of microwave heating, some specimens of Al_2O_3 ceramics are sintered under various sintering temperatures by a conventional heating method and a hybrid microwave heating technique. The heating velocity of microwave and conventional methods, the rate of volume change, bulk density, water absorption and apparent porosity of the conventionally and microwave sintered samples were investigated, and the experimental results of two kind samples were compared.

The following conclusions can be made via the comparisons of the experimental results of two kind samples.

(1) Microwave sintering method has the characteristic of rapid heating compared to conventional method.
(2) The sinterability of hybrid microwave sintered sample is better than that of conventionally sintered sample at the same sintering temperature.
(3) Microwave sintering method can reduced the sintering temperature of ceramics when compare with conventional method.

In Chapter 6, to evaluate the homogeneous performance of the microwave sintered ceramics, Al_2O_3 ceramics specimens are sintered by hybrid microwave and conventional sintering method, respectively. The behaviors of thermal residual stress and the pore ratios were investigated, and utilized to characterize hybrid microwave sintered specimens and conventionally sintered specimens. The characteristics of two-directional hybrid microwave sintering were also confirmed by using thermal residual stress and the pore ratio distribution. Otherwise, the Vickers Hardness (HV) distribution was also investigated. The advantages of the performance of hybrid microwave sintered ceramics are evaluated on a microscopic scale.

The following conclusions can be made:

(1) A correlation between the thermal residual stress and the pore ratio in the composite ceramic material is confirmed, and it is possible to evaluate the sinterability of ceramics from thermal residual stress measurements.

(2) The uniform heating property of two-directional hybrid microwave sintering is confirmed by the thermal residual stress and the pore ratio investigations on a microscopic scale.

(3) It is confirmed that sinterability of ceramics is improved by hybrid microwave heating, and Vickers hardness tests on microwave sintered specimens revealed superior mechanical properties compared to conventionally sintered specimens.

Finally, the conclusions obtained through these studies are summarized in Chapter 7.

As mentioned above, this thesis evaluates the strength characteristics of fine grained steel and microwave sintered ceramics under various processing conditions or environment by using the X-ray diffraction technique. The results obtained in this study are expected to be contributive to promoting the study on fine grained steel and microwave sintered ceramics.

学位論文審査結果の要旨

平成 20 年 7 月 30 日第 1 回学位論文審査委員会を開催し，提出された学位論文及び関係資料に基づき論文内容を詳細に検討した．さらに，平成 20 年 7 月 30 日に行われた口頭発表後に，第 2 回学位論文審査委員会を開き，協議の結果，以下のように判定した．

本論文は新世代の高強度材料である結晶粒微細化材料についてミクロな視点から X 線により材料の強度評価を行ったものであり，特に微細粒鋼およびマイクロ波焼結したセラミックスについて検討を行ったものである．本研究ではまず微細粒鋼 NFG600 と圧延鋼 SM490 に着目し，研削・エメリー研磨による有向性加工後の残留応力を X 線応力測定法で測定し，三軸応力解析を実施し残留応力分布に及ぼす加工方法および結晶粒径の影響を明らかにした．加えて，その両材を用いて応力腐食割れ発生実験を行い，き裂発生寿命におよぼす結晶粒径の影響について検討を行った．また，マイクロ波焼結と従来の電気炉焼結方法によってそれぞれアルミナセラミックス試験片を焼結した．半径方向と深さ方向の残留応力分布および気孔率分布を調べ，ビッカース硬さを測定し，両方法による試験片を比較した．上記した微視的な視点からの実験より，マイクロ波加熱の内部加熱の特徴を明らかにし，焼結性改善への提案を行った．以上の内容は，今後の超微細粒鋼開発およびマイクロ波によるセラミックス焼結技術に大きく貢献できるものと確信する．

以上，本論文は博士（工学）の学位論文に値するものと判定する．