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授 与 学 位	博　士（工 学）
学位授与年月日	平成 4 年 3 月 27 日
学位授与の根拠法規	学位規則第 5 条第 1 項
研究科，専攻の名称	東北大学大学院工学研究科 （博士課程）材料加工学専攻
学 位 論 文 題 目	Microstructure and Superconducting Properties of Plastically Deformed $YBa_2Cu_3O_{7-x}$ （酸化物超伝導体 $YBa_2Cu_3O_{7-x}$ の塑性加工による組織制御と超伝導特性）
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論 文 内 容 要 旨

Chapter 1　Introduction

This chapter describes the background of Tc and Jc impovement in conventional superconductors and oxide superconductors. The characteristics of crystal structure and microstructure of high Tc superconductors are reviewed for better understanding of deformation behavior in oxide superconductors. Major focus is on fabrication methods of oxide superconductors to obtain highly oriented microstructure. After outlining relevant studies to fabrication techniques and theories of oxide superconductors, the purpose, necessity and significance of this research are described. In essence, main objective of this study is to develop new processes to produce the optimized microstructure for high Jc. For this purpose, fundamental studies are performed on plastic deformation by high temperature compression. Also, microstructural changes during deformation are investigated by optical microscopy, scanning electron microscopy (aided by ECP methed) and transmission electron microscopy. Based on the obtained results improvement of Jc properties is discussed.

Chapter 2 High Temperature Deformation Behavior of $YBa_2Cu_3O_{7-x}$

The high temperature deformation behavior of $YBa_2Cu_3O_{7-x}$ polycrystals was studied as a function of temperature, strain rates and grain sizes. Stress-strain curves are presented for $YBa_2Cu_2O_{7-x}$ polycrystals with grain sizes of 3 μm and 7 μm at $1.3\times10^{-5}s^{-1}$ at temperatures from 1136 to 1253K. Steady state deformation appeared above 1203K for both samples. The plots of the logarithm of steady-state stress as a function of T^{-1} for both grain sizes above 1203K gave a linear relation. From the slope of the straight lines, activation energies are determined to be approximately 150kJ/mol independent of grain size. The strain rate sensitivity of steady-state stress at various initial strain rates between 1.3×10^{-5} and $4\times10^{-4}s^{-1}$ in the temperature range 1203 to 1243K gives a stress exponent of 1.3. Dislocations were rarely observed in the samples compressed above 1203K. On the other hand, work-hardening appeared in stress-strain curves below 1203K and TEM observations revealed that dislocations with a high density were introduced during deformation.

The obtained results on the compression tests and microstructural observations suggest that the dominant deformation mechanism is diffusional creep associated with grain boundary sliding above 1203K and the dislocation glide accompaniced with grain boundary sliding below 1203K. Finally, the grain growth is enhanced by deformation and is accompanied with development of strong anisotropy in grain shape and preferred arrangement.

Chapter 3 Analysis of Orientation Distribution in $YBa_2Cu_3O_{7-x}$ Polycrystals by Electron Channeling Patterns

The analysis of orientation distribution by electron channeling patterns in a small-sized $YBa_2Cu_3O_{7-x}$ tape sample was performed to investigate the applicability of this method for oxide superconductors. The standard channeling map for $YBa_2Cu_3O_{7-x}$ superconductors in the range of a stereographic quadrant was produced for first time. Orientation distribution of grains in pressed tapes for Jc measurements was analyzed by taking ECPs for individual grains as a function of reduction (tape thickness). In the 60 μm thick tape which is heavily pressed, (001) texture is considerably strong and (001) planes of most grains are inclined within 30° to the specimen surface. In the 200 μm thick tape, (001) texture is weak in comparison to the 60 μm tape. No apparent preferred orientation is observed in the as-sintered pellet. High Jc in pressed $YBa_2Cu_3O_{7-x}$ polycrystals is ascribed to (001) texture. Pole figures by X-ray technique are in excellent agreement with the results of ECP analysis. It is concluded that the ECP technique is very useful for the analysis of orientation distribution in small sized specimens such as tapes or thin films.

Chapter 4 Microstructure Control of $YBa_2Cu_3O_{7-x}$ by High Temperature Compression

The texture development during high temperature deformation under optimum deformation condition was examined by simple uniaxial compression and compression with grooved dies. The optimum deformation condition was determined by compression tests at temperature 1203K and strain rate $1.3\times10^{-5}s^{-1}$. At this selected optimum condition, the texture developed very strongly by uniaxial compression and compression with grooved dies. It was apparent that grains in the compressed sample concentrated strongly on the center of the (001) pole figure. The deviation angle of the (001) plane in each grain from the sample surface is almost within 30° for both samples. In uniaxial compression and compression with grooved dies, 70% and 85% grains of all the measured grains were within deviation angle of 25° , respectively. In compression with grooved dies a- or b-plane texture was developed. The transport critical current density $J_{c(trans)}$ at 77K was 450 A/cm² for the sample compressed 50% uniaxially and 500A/cm² for sample compressed with grooved dies, indicating that the $J_{c(trans)}$ values are nearly one order higher than that of the as-sintered sample. The intragrain critical current density $J_{c(trans)}$ at 4.5K in a magnetic field parallel to compressin axis was 2.4×10^5A/cm² for the sample compressed 50% uniaxially and 3.8×10^5A/cm² for the sample 50% compressed with grooved dies. The low $J_{c(trans)}$ is attributed to microcracking induced by anisotropic contraction of grains after reoxidizing.

Chapter 5 Microstructure Control and Critical Current Density of $YBa_2Cu_3O_{7-x}$-Ag Composites by High Temperature Compression

The effect of Ag_2O adition on microcracking induced by phase transition and reoxidizing heat treatment of a $YBa_2Cu_3O_{7-x}$ polycrystal was investigated. Jc was measured on compressed samples with various amounts of Ag_2O as a function of plastic strain at 77K. The specimens doped with 20 and 30w/o Ag_2O are well deformed to 55% plastic strain in the low stress level compared to the sample without Ag_2O. And reoxidized sample after 50% uniaxial compression showed very strong (001) reflections of the $YBa_2Cu_3O_{7-x}$ phase. Uniformly distributed Ag particles were found to play an important role in relieving the anisotropic thermal stress in the $YBa_2Cu_3O_{7-x}$-Ag composites. The maximum $J_{c(trans)}$ value was obtained from the sample doped with the 20w/o Ag_2O. The $J_{c(trans)}$ value at 77K and at OT was 1400 A/cm². The agglomerations of Ag particles were formed after compression in the sample doped with 30 w/o Ag_2O containing sample. This phenomenon reduces the path of the current flows and eventually decreases $J_{c(trans)}$.

Chapter 6 Summary

The major experimental results, findings and conclusions obtained from this doctoral research are summarized in this chapter. The overall conclusion is drawn and presented and future research areas are suggested.

審査結果の要旨

実用超伝導体の臨界電流密度は組織に影響されることが知られている。

本論文は，高い超伝導転移温度をもつ酸化物超伝導体 $YBa_2Cu_3O_{7-x}$ の臨界電流密度の向上を目指して，高温塑性加工による組織制御を検討し，さらに組織と臨界電流密度との関係を調べた結果をまとめたもので，全編6章よりなる。

第1章は緒論であり，本研究の背景と目的を述べている。

第2章では，$YBa_2Cu_3O_{7-x}$ の高温圧縮変形挙動について調べている。1203K以上の温度で初期ひずみ速度$1.3\times10^{-5}s^{-1}$の圧縮変形を行うと定常変形が現れ，延性的な圧縮変形が可能であること，このときの応力指数は1.3であり，変形組織には転位がほとんど観察されないことを見出し，変形は拡散クリープにより律速されていることを明らかにしている。

第3章では，組織因子の中でも臨界電流密度に関係すると考えられる結晶粒方位分布を変形後の微小形状の試料で測定するために，新たにエレクトロン・チャンネリング・パターン（ECP）を用いた $YBa_2Cu_3O_{7-x}$ の結晶粒方位分布の解析法を開発した。本法の有効性を実証するとともに，室温加工した標準試料で（001）集合組織の発達により臨界電流密度が上昇することを示している。

第4章では，高温一軸圧縮加工した $YBa_2Cu_3O_{7-x}$ の結晶粒方位分布を ECP 法で測定し，c軸に垂直な面が圧縮軸に垂直にそろうとともに，異方性のある形状に粒成長し，それにともなって粒界電流密度が上昇することを明らかにしている。また側面拘束で圧縮することにより，c軸方位ばかりでなく，aまたはb軸が，拘束された側面に平行にそろうことも示されている。

第5章では，高温加工性を改善し，さらに加工後の酸素中熱処理によるマイクロラック導入するため，銀粒子を微細に分散させた $YBa_2Cu_3O_{7-x}$ を作製し，その加工性，粒界電流密度が向上することを示している。

第6章は総括である。

以上要するに本論文は，$YBa_2Cu_3O_{7-x}$ の延性的な塑性加工条件を明らかにした上で，塑性加工により組織制御が可能であることを示し，さらに臨界電流密度を向上させるための組織制御に対して指針を与えたもので，材料加工学の発展に寄与するところが少なくない。

よって，本論文は博士（工学）の学位論文として合格と認める。