Heterogeneous Deformation Processes in Novel Iron Based and Copper Based Alloys

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論文内容要旨

Plastic deformation of polycrystalline metals is heterogeneous due to mutual interaction of grains having different crystallographic orientations, as described by a classical polycrystal deformation model by M. F. Ashby. Predicting and characterizing the heterogeneous deformation require knowledge of deformation mechanisms. Plastic deformation of polycrystalline metals is generally explained by three deformation mechanisms, namely slip, twining, and martensitic transformation, which compete with each other and play decisive role in determining material properties. Martensitic transformation has been intensively studied during the past decades because it is fundamentally associated with some interesting phenomena such as shape memory effect and superelasticity in several metals. For example, Ti-Ni and Cu-Al-Mn alloys that transform martensitically show shape memory and superelastic behavior, and thus they are commercially used as shape memory and superelastic materials. Martensitic transformation is also of great technological importance in steels where outstanding strength and ductility are required. The concept of martensitic transformation was originally introduced to describe hard microconstituent phase formed in quenched conventional carbon steels. In current transformation induced plasticity (TRIP) steels, classified to advanced steels with exceptional combination of high strength and good ductility, the martensitic transformation induced by deformation is important. In these steels, plasticity is assisted by strain induced martensitic transformation and it explains their characteristic mechanical properties. Since above-mentioned unique phenomena caused by martensitic transformation are not obtainable in usual metals, we refer them as “novel” phenomena, and the related alloys as “novel” alloys. Our study focused on the deformation process including martensitic transformation in novel iron based and copper based alloys such as Cu-Al-Mn superelastic alloy, C-Mn-Si TRIP steels, and Fe-Mn-Si-Cr shape memory alloy (SMA).

To characterize plastic deformation and related deformation mechanisms in polycrystalline metals, it is useful to use local scale characterization techniques. For the characterization of structural heterogeneity at the local scale, modern experimental tools such as electron backscattering diffraction (EBSD) and white X-ray microbeam diffraction has been increasingly used. This local scale characterization allows mapping of the crystalline orientation distribution on the sample surface or sample volume and thus provides significant information on local strain and stress. These techniques are considered to be particularly useful for characterization of heterogeneous deformation processes in polycrystalline materials and also for analysis of their complex microstructure.

In the present study, these techniques were mainly used for local microstructural analyses in novel iron based and copper based alloys with specific analysis purposes, including grain boundary constraint and heterogeneous deformation characterization in a Cu-Al-Mn superelastic alloy, quantification of bainite phase in TRIP steels, and characterization of orientation dependence of martensitic transformation and residual stress evolution in a Fe-Mn-Si-Cr SMA. Above-mentioned analyses are fundamentally important to understand the origin of unique mechanical properties in each alloy system. This thesis consists of 5 Chapters, and essential points of every Chapter are summarized as follows.

In **Chapter 1** 「**Introduction**」, white X-ray microbeam diffraction and EBSD used for mesoscale (local scale) microstructural characterization are introduced. Also, their possible applications in Cu-Al-Mn superelastic alloys, Fe-Mn-Si-Cr SMA, and TRIP steels are introduced.

White X-ray microbeam diffraction is a recently emerged powerful tool for measuring internal stress/strain evolution in polycrystalline materials. This technique allows us to study intragranular strain variation and can be utilized as a new microstructure characterization technique. A visualization technique developed at BL28B2 beamline of SPring-8 in Japan was effective in visualizing grain boundaries and inside the grains. This technique is capable of simultaneous strain measurement under load conditions, and thus offers a unique opportunity for comprehensive deformation mechanism and internal strain/stress variation characterization in polycrystalline materials.

EBSD is a materials microstructural-crystallographic characterization technique to mainly measure crystallographic orientation. In the last decade, EBSD has found wide scale applications in microstructural characterization of multi-phased steels such as transformation induced plasticity (TRIP). The applications of EBSD to these types of steels may include the identification of specific phases on their complex microstructure in which the phases are not detectable by usual methods. Recently, new method using image quality metrics in EBSD was developed to study more accurately the complex microstructure. In this study, EBSD band slope (BS) parameter was utilized to quantitatively measure constituent phases in the microstructure of TRIP steels. Meanwhile, EBSD also can be used to study orientation dependence of martensitic transformation in Fe-Mn-Si-Cr SMA. Shape memory effect of SMA is closely related to grain orientation because martensitic transformation depends on grain orientation. Therefore, the measurement of martensitic transformation in individual grain with different orientation is fundamentally important to understand shape memory effect; however, the martensitic transformation behavior in specific grains with different orientations has not been systematically explored by using the EBSD. In this study, formation of martensite was examined in grains with distinct orientation by using EBSD in order to investigate the relationship between the crystallographic orientation of grains and martensitic transformation which is strictly related to SME.

In **Chapter 2** 「**Unique stress distribution in a superelastic Cu-Al-Mn alloy characterized by white X-ray microbeam diffraction**」, grain boundary constraint and inhomogeneous deformation process in Cu-Al-Mn alloy studied by white X-ray microbeam diffraction is explained. Grain boundary constraint is an important factor determining superelastic properties in the Cu-Al-Mn alloy. The superelastic properties in the alloy were reported to strongly depend on microstructural parameters such as grain size, constituent phases, and texture. For example, increase in grain size relative to specimen thickness enhances superelastic strain. The coarse grain results can be attributed to relaxation of grain boundary constraint. Direct experimental observation of grain boundary constraint would be possible by measuring local strain/stress evolution within a grain using white X-ray microbeam diffraction. In this study, the grain boundaries constraint effect on lattice strain/stress was evaluated by comparing the evolution behavior of lattice strain at the grain boundary to the center. Grain boundary constraint was demonstrated by the lattice strain analysis showing the formation of large compressive lattice strain and large residual lattice strain near grain boundary regions. Grain boundary constraint was also revealed by observing formation of large compressive stress at grain boundary region within a grain due to interactions with neighboring grains with different orientation.

Stress analysis and Laue spot streaking analysis using white X-ray microbeam diffraction can give fundamental information on the heterogeneous deformation. Microstructure of an investigated tensile sample was imaged using recently developed visualization technique to determine stress analysis position. Using the technique, very clear grain boundary image was obtained for the first time. In addition, comparison of the visualized image with an image of the same area obtained by EBSD technique revealed that they are almost comparable in terms of their grain boundary structure, confirming the validity of the visualization technique. Stress was measured on several local points (grains) in the Cu-Al-Mn alloy under load corresponding to 8% strain and after subsequent unloading. The results revealed inhomogeneous stress evolution by deformation. Laue spot streaking arises due to the presence of strain and orientation gradient, and thus microstructural homogeneity can be evaluated by observing evolution of Laue spot streaking in the microstructure. To study orientation deviation caused by martensitic transformation, we quantitatively measured Laue spot streaking within a single grain and also in several grains. The results revealed that the degree of Laue spot streaking differed according to position within a grain. This is attributed to the formation of locally different amount of martensite within the grain, which results in evolution of inhomogeneous orientation gradient. Degree of Laue spot streaking also depended on grain orientation in tensile direction. The grain with large Schmid factor for martensitic transformation showed pronounced Laue spot streaking. The observation on the heterogeneous evolution of Laue spot streaking within a single grain and at grain scale clearly demonstrated heterogeneous deformation behavior of this superelastic alloy.

In **Chapter 3** 「**Microscopic distribution of multi-phases in deformed TRIP steels determined using advanced EBSD analysis**」, determination of bainite phase in TRIP steels using EBSD BS parameter is explained. TRIP steels are characterized by multiphase microstructure composed of ferrite, bainite, and retained austenite. Although there have been many studies on the analysis of the complex microstructure of TRIP steels, bainite is still regarded as a phase which is difficult to detect using usual microstructural characterization method, such as optical microscopy. The use of EBSD BS parameter is an alternative and advanced method in measuring bainite quantitatively. The BS is determined by quality of Kikuchi patterns and is physically related to crystallographic uniformity of material. Quality of Kikuchi patterns, i.e., BS, is sensitive to defect density and surface feature. We found that bainite showed much lower BS value as compared to the other phases because of its high defect density and very rough surface. Based on this fact and by assigning proper BS threshold value, bainite was successfully separated from other phases. Further, bainite was quantitatively measured in several samples austempered for different time. The results revealed that while volume fraction of retained austenite decreased with increasing austempering time, the volume faction of bainite increased. This is because retained austenite gradually transforms into bainite during austempering process at bainitic transformation temperature range (430°C).

In **Chapter 4** 「**Orientation dependence of stress-induced martensitic transformation and evolution of residual stress in Fe-Mn-Si-Cr alloys studied by EBSD and white X-ray microbeam diffraction**」, martensitic transformation in individual grains with different orientation studied by EBSD and residual stress evolution studied by white X-ray microbeam diffraction is explained. For this study, two different types of samples were prepared; (1) sheet type SMA prepared by conventional furnace melting and hot/cold rolling and (2) cast type SMA prepared by centrifugal casting. Sheet type SMA was used to study orientation dependence of martensitic transformation and cast type SMA was used for stress analysis because it contains coarse grain. EBSD, as an effective local microstructural characterization technique, can be used to observe deformation process in the desired area in polycrystalline materials. However, there has been almost no systematic study to observe martensitic transformation in individual grains in Fe-Mn-Si-Cr SMA with EBSD for the purpose of characterizing orientation dependence of martensitic transformation. Martensitic transformation was observed in three grains with different orientation (near <101>, <001>, and <111>//tensile direction (TD)) in sheet type SMA using EBSD to investigate relation between grain orientation and martensitic transformation. The results showed that as compared to <001> and <111>//TD grains, large amount of martensite was formed in <101>//TD grain. This is explained by the fact that <101>//TD grain has higher Schmid factor of 0.47 than those for <111> and <001>//TD grains which are 0.31 and 0.23, respectively. To investigate shape memory behavior and relation between residual stress and shape memory effect, evolution of residual stress in two cast type SMA samples containing coarse grains with near <001> or <144>//TD orientation was measured using white X-ray microbeam diffraction. Although <144>//TD sample showed larger recovery strain than that of <001>//TD sample, residual stress after recovery in both samples was found to be very small, and no significant difference in the magnitude of residual stress was found between the samples. This indicates that stress in these samples evolved by deformation mostly released upon subsequent recovery heating as a result of shape recovery. Laue spot streaking was measured in <144>//TD sample during the sample memory behavior. Significant Laue spot streaking was found after tensile deformation to 23% strain, indicating formation of orientation gradient and internal strain/stress. After recovery heating, the streaking mostly disappeared although small amount of it still remained probably due to orientation rotation in subgrains. This reversible behavior of Laue spot streaking explains overall shape memory behavior of the Fe-Mn-Si-Cr SMA.

In **Chapter 5** 「**Conclusions**」, the conclusions drawn from above works are summarized.

As shown in this thesis, stress analysis and Laue spot streaking analysis based on white X-ray microbeam diffraction can give much information on heterogeneous deformation process at local scale. Particularly, the observation on the heterogeneity of the Laue spot streaking within a single grain and at grain scale clearly demonstrated heterogeneous deformation behavior of the superelastic alloy. It is concluded that comprehensive study by *in situ* observation on the evolution of microstructure, strain/stress, and Laue spot streaking using white X-ray microbeam diffraction is an effective means of heterogeneous deformation characterization. Meanwhile, the use of EBSD BS parameter was found to be effective in identification and quantification of bainite in complex microstructure of TRIP steels. As the BS parameter is sensitive to defect density, it is considered to be used to study deformation characteristics in various materials as well as their complex microstructure.

論文審査結果の要旨

鉄合金や銅合金の中には、形状記憶効果、超弾性、変態誘起塑性を示す合金があり、これらの特異な力学特性にはマルテンサイト変態およびその逆変態が関係している。本研究では、これらの特徴的な変形挙動を示す新規の鉄合金や銅合金における変形過程を明らかにするために、放射光を用いたマイクロビーム白色X線回折法、電子後方散乱回折法等を用いて、微視的な不均一変形過程の解析を行った。

本論文においては、まず超弾性を示す代表的なCu-Al-Mn多結晶合金について、粗大結晶粒内の格子歪を、マイクロビーム白色X線による透過ラウエパターン測定により調べた。測定においては、二次元的なラウエパターンのマッピングを行うとともに、幾つかのラウエ斑点に対してエネルギースペクトルを測定し、各回折面における格子歪を評価した。これらの結果から、粗大結晶粒内で生成するマルテンサイト変態の結晶方位による違い、結晶粒中心部や粒界付近などの測定箇所における応力状態の違い等を明らかにした。これらの結果は電子後方散乱回折法による結果とも対応付けて、各結晶粒における幾何学的な変態歪や主応力等について考察した。

次に、変態誘起塑性を示す低合金鋼（TRIP鋼）について、オーステナイト相とフェライト相が共存する温度領域で焼鈍した後にオーステンパリングを行い、試料の残留オーステナイト相等の微細組織と力学特性との関係等を調べた。微細に分散する相の解析等には電子後方散乱回折法を用い、特に、オーステンパリング中に生成するベイナイト相の量を評価する方法を確立した。この方法により、これまでフェライト相と区別することが困難でベイナイト相の量が熱処理によって系統的に変化することを明らかにし、これらの結果から微視的な相分布と力学特性の結果との関係を明らかにすることができた。これらの手法は、新たな組織解析法としても重要であると言える。

さらに、鉄系形状記憶合金のFe-Mn-Si合金について、室温での変形およびその後の加熱による形状回復過程（形状記憶効果）を、構造変化と関係づけて詳細に調べた。変形量を変えた試料の形状回復率評価および電子後方散乱回折による解析から、形状回復率がマルテンサイト変態相の量や多結晶の集合組織と関係することを示した。また、異なる方位のほぼ単結晶試料の引っ張り変形挙動とマルテンサイト変態の割合や形状記憶特性の関係を調べ、一種類のマルテンサイト変態が優先的に起こる方位を持つ結晶粒で、大きな形状回復率をもつことを示した。これらの結果は、これまで不明瞭であった形状回復率への多結晶の影響を明らかにした結果として重要である。

以上の研究成果は、投稿論文6件(第一著者：3件)、国際会議2件で発表され、高い評価を受けた。したがって、本論文は博士論文として充分な新規性および進歩性があり、金属フロンティア工学の発展への寄与が少なくない。

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。