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学 位 論 文 題 目	**Development of high strength bimodally grained Fe- and Al-based nanocomposite materials** （Fe 基および Al 基 2 粒子サイズ分散高強度ナノコンポジット材料の開発）

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論 文 の 内 容 の 要 旨

Even though nanocrystalline materials exhibit very high strength, the ductility is either low or completely absent due to the early onset of plastic instability originating from the lack of dislocation activities at such length scales. In this work. we attempted to achieve both high strength and good ductility in achieving bimodally grained nanostructure with ultrafine dispersion of oxide particles. The thesis comprises of 4 chapters. Chapter l expalins the background of the research. and gives literature review of previous investigations to optimize strength and ductility in different length scales, *i.e.*, micrometer, sub-micrometer and nanocrystalline materials. The aim of this study was to achieve a high strength and good ductility in Fe- and Al- base materials by designing the microstructure which consists of bimodal grain size distribution (nano/ultrafine grain and coarse grains) with nano precipitates. The bimodal grain size distribution and nano precipitates were used to improve the ductility and nano-grain sizes were used to achieve high strength. Chapter 2 describes the detailed experimental techniques used for the preparation of nanocrystalline powders, consolidation and subsequent characterization. Chapter 3 reports the results and discussions on the mechanical properties and microstructure characterization of mechanically milled Fe. Chapter 4 describes a detailed investigation on developing high strength in Al-Zr and multicomponent (Al-Cu-Si-Mn-Ti-Zr) alloys.

Mechanical milling/alloying was employed to obtain powders with nanosized grains. The materials used in this study were Fe (99+%), Al (99.99%), Zr (99.9%), Si(99.99%), homemade intermetallic phases of AK_3Zr, AL_6Mn, Al_2Cu and Al_8Ti. The resulting powders were consolidated by the spark plasma sintering (SPS)process and the typical dimension of the sintered samples was 5 mm thickness and 10 mm diameter. From the sintered samples, rectangle compression specimens of 2 mm × 2 mm × 4 mm and rectangle dog-bone tensile specimens with 2 mm gage length, 0.5 mm width and 0.5 mm thickness were prepared. These specimens were tested in an Instron machine with a strain rate of l × 10-4 and l × 10-3 s-1. The microstructural characterization of the samples was performed by x-ray diffractometry (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and 3 dimensional atom probe (3DAP) techniques.

Bimodally grained nanocrystalline Fe consisting of nanograined, coarse grained and uniformly dispersed nanosized oxide particles were processed by mechanical milling of pure Fe powder and subsequent spark plasma sintering. A compressive strength of 3 GPa with 5% plasticity was achieved and by changing the sintering temperature a high tensile strength of 2.1 GPa with 5% elongation was realized. Although the nanograined region is also responsible for the high strength. half of the strength in the current samples can be attributed to the precipitation hardening of nanosized oxide particles. The yield point elongation observed in the tensile test suggests the presence of interstitial carbon in the coarse grained regions. In the case of nanocrystalline grains, carbon is segregated at the grain boundaries. The excellent stability of nanograins even after a sintering temperature of 790℃ is considered to be due to the presence of carbon segregation and oxide particles at the grain boundaries and at triple junction, which effectively pin the grain boundaries and restrict grain growth. Since excellent balance of high strength and ductility can be achieved by optimizing sintering and subsequent heat treatment conditions. the mechanical milling and sintering route of pure Fe may be a promising way to develop high strength nanostructured steel.

Three different processing routes were explored to develop Al-Zr nanocomposite alloys using mechanical alloying and spark plasma sintering methods. Depending on the route of milling adopted, the powder in the as-milled condition consisted of either a solid solution of Zr in Al or a mixture of Al solid solution and Al_3Zr (LI2)phases. The alloys after sintering consisted of Al and Al_3Zr (Ll_2)with grain sizes of less than 100 nm. These nanocomposite alloys exhibited a high compressive strength of l GPa with 10% plasticity. The high strength observed in these alloys was explained on the basis of the retention of nanometer sized grains and also the fine dispersion of the Ll_2, phase. On the other hand. the good amount of plasticity was explained to be due to excellent bonding between the powder particles and the presence of coarse Al grains in the matrix.

審 査 の 結 果 の 要 旨

本論文は純鉄粉ならびにA1合金をメカニカルミリンダでナノ結晶化した後に、スパークプラズマ焼結法でバルク体に成形し、その微細組織と力学特性を評価した研究結果をまとめた学術研究であり、下記の２点から高く評価される。(1) 純鉄でナノ組織を制御することにより、最高3GPaという驚異的な強度を達成した。焼結条件を制御することにより、ナノ組織に粗大結晶粒を適度に分散させることにより超高強度と引っ張り延性の両者を両立できることを実験的にしめした。(2) これらのユニークな力学特性を有する材料の微細祖織を精密に解析することにより、力学特性発現のメカニズムを解明した。ナノ結晶Feの力学特性として世界最高レベルであり、強度・延性の両者をバランス良く達成する手法を提案したことから、極めて学術的価値の高い論文と判断される。

よって、著者は博士（工学）の学位を受けるに十分な資格を有するものと認める。