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발표 예정 논문

Preparation and applications of bulk cubic RFe_2 Tb-Dy-Fe magnetostricitve single crystals

Abstract

Magnetostriction is the change in any dimension of magnetic material caused by a change in its magnetic state. Since the discovery of large magnetostrictive strains in dysprosium, terbium and their alloys with iron, attention has been given to $Tb_{0.3}Dy_{0.7}Fe_2$ which is highly magnetostrictive material for applications by virtue of its high Curie temperature and in some cases, lower magneto-crystalline anisotropy, suitable for devices at room temperature with relatively samll applied field. It has been the object of a series of experiemental studies since it is a desirable candidate for use in commercial transductive devices, ultrusonic transducers, microstoning devices, as well as smart materials etc. As the magnetic anisotropies are encountered in this special alloy, grain-orientation crystal or single crystal is essential for achieving high magnetostriction. It is known that single crystal $Tb_{0.3}Dy_{0.7}Fe_2$ can be obtained by an induction free standing melt zone (FSMZ) or modified Bridgman-Stockbargers method, however, the contamination and twin structure make it difficult to get high-qualit single crystals.

Bulk $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ single crystal has been prepared, completely without contimination, by Hukin' s magnetic levitation cold crucible in Czochralski techniques during our researches. The starting materials, Tb (99.95% wt), Dy (99.98%wt) and Fe (99.96% wt), are used to synthsize the prescursor alloy. The weight of each loading is generally 120-160g. Seed crystal can be obtained by using the neck-in and elimination method with a pure iron bar (1mm in diameter) at the very beginning of crystal growth. Single crystal, growth with this seed crystal of $[11\bar{2}]$, can eliminate the twins which are usually existing in Terfenol-D and show a

high magnetostrictive performances. X-ray diffraction analyses demenstrates that the single crystal is pure cubic Laves RFe_2 phase with $Fd3m(O_h^7)$ structure. Elements composition researches are carried out by the combination of the chemical analyses, elctron microprobe analyses and atom absorbspectral analyses. Microstructures are studied by X-ray diffraction and electron scan microscope analyses. Results of growth conditions and single crystal analyses are shown in the following:

1)Pulling rate	0. 1—10mm/min。
2)Rotaion rate	30—50r/min。
3)P/R	<1/5KW。
4)Power supply	15—20KW。
5)Inducing F.	100—120KHz
6)Size	Φ 10—15mm。
	L 120—150mm。
7)Weight	80—100g。
9)Direction	$[11\bar{2}]\pm 1°$。
10)	$\lambda_S = 1950$ ppm(10MPa)
11)	$d_{33} = 2.0$ppm/Oe。
12)	$K_{eff} = 0.89$。
13)	E=67. 3GPa
14)	$\sigma_b = 323.5$MPa。

With this high performance single crystal, prototype of microstoning device with a high displacement of 25 μm has been designed and developed also during our past reseaches. And it is the first time to find that there is the superlattice structure in the cubic cell of $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ single crystal.

It has been proved that the elmination of twins in $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ single crystal is a significant advance in magnetostrictive materials researches, however, the efforts for growth single crystal of [111] has not succeeded. It is believed that there is a long way (as well as an interesting way) to go in the preparation and applications of $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ magnetostictive single crystals in the coming future。

摘　　要

立方相 RFe_2 Tb-Dy-Fe 超磁致伸缩材料是国外 80 年代初发展起来的一类具有室温超大磁致伸缩效应($\lambda_s \geqslant 1500ppm$)的新型稀土-过渡族元素金属间化合物功能材料。可广泛应用于声纳，雷达，高精度位移控制，机电转换，超微测量和加工等数十个高技术领域。多年来，材料制备过程中难以避免的污染，以及材料本身的磁各向异性等问题，一直是困扰和限制这类材料的商品化生产和性能提高的主要障碍。迄今为止，国外仅有美国，瑞典的几家公司可以从事以 Terfenol-D 为代表的 Tb-Dy-Fe 超磁致伸缩材料的生产和应用研究。但由于其产品主要为定向结晶(孪生单晶)材料，第二相和孪晶界的影响，导致 Terfenol-D 实际的磁致伸缩性能与理论值相比尚有较大差距。国内虽然很早就开展过 Tb-Dy-Fe 多晶材料的探索，但始终未能有所突破。

本研究在总结前人工作的基础上，采用提拉法无污染磁悬浮冷坩埚晶体生长技术，独特的晶体成份设计、相组成控制、晶体加工和处理工艺，制备出具有当今国际先进水平的大尺寸、立方相 RFe_2 $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ 单晶，其主要技术指标：

尺寸： 最大直径	Φ	10－15mm。
最大长度	L	120－150mm。
最大重量	W	80－100g。
生长方向	〔$11\bar{2}$〕	偏差 ±1°。

室温饱和磁致伸缩系数	$\lambda_s = 1950ppm(10MPa)$。
室温饱和磁致伸缩率	$d_{33} = 2.0ppm/Oe$。
有效机电耦合系数	$K_{eff} = 0.89$。
杨氏模量	$E = 6.73 \times 10^{10} N/m^2$。
抗压强度	$\sigma_b = 325.3MPa$。

单晶不仅消除了以往定向结晶材料中孪晶结构的影响，而且成功地控制了 RFe_2 组织中的富稀土相含量，其主要性能指标明显优于目前国外普遍使用的具有类似成份的 Terfenol-D 定向结晶材料。并

已能实现小批量生产，达到实用化研究水平。

通过本课题的研究工作，初步形成了一套全新的 Tb-Dy-Fe 合金单晶材料的成份设计和相控制的理论体系，解决了 Tb-Dy-Fe 单晶由于 X-ray 吸收因子过高而无法采用常规实验方法定向的难题，建立了包括电子探针、化学分析、光谱分析在内的 Tb-Dy-Fe 合金体系的综合成份分析方法，提出了提拉法冷坩埚生长 Tb-Dy-Fe 单晶的工艺控制参数：

拉速/转速＜1/5。

拉速　0.1－10mm/分。

转速　30－50 转/分。

摸索出特殊的 $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ 单晶的线切割加工和二次退火热处理工艺. 并成功地研制出国内第一台超磁致伸缩 μm 级微位移致动器原型，完成了 Tb-Dy-Fe 单晶材料从成份设计、工艺控制、晶体生长、性能分析到材料应用的一整套研究和应用探索过程。

本研究工作中，采用 X-ray 结构分析技术，首次发现了 $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ 单晶中存在超结构现象。这一发现，为进一步研究晶格内部原子排布与单晶性能之间的关系，揭示 Tb-Dy-Fe 超磁致伸缩材料的微观磁化机理，提供了一个新的探索途径。

本研究工作的开展，结束了我国没有高性能、大尺寸立方相 RFe_2 Tb-Dy-Fe 超磁致伸缩材料的研究和应用的历史，填补了我国在此高技术新材料领域的一项空白。对于新一代高性能大尺寸立方相 RFe_2 Tb-Dy-Fe 超磁致伸缩单晶材料的大规模生产和应用进行了有意义的尝试。