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Report:

Soft magnetic composites with enhanced performance and their key production technologies

Mi YAN[†], Chen WU
(*State Key Laboratory of Silicon Materials, School of Materials Science and Engineering, Zhejiang University, Hangzhou 310027, China*)
[†]E-mail: mse_yanmi@zju.edu.cn

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Soft magnetic composites (SMCs) contain metallic magnetic powders embedded in an insulation matrix, and are usually prepared by procedures including powder production, insulation coating, binding, compaction, and annealing. They have attracted extensive interest as fundamental electric and electronic components in the fields of energy, information, transportation, and national defense. With the development of electric and electronic devices with properties suitable for high frequency, large power, energy saving, and being electromagnetically compatible, the demand and requirements for SMCs become significantly higher. The SMC industry in China used to lag far behind overseas competitors with a huge technological gap. The main domestic products were Fe SMCs with only a small amount of alloy SMCs. Such SMCs exhibited low magnetization and unsatisfactory DC-bias properties with double the loss compared to the overseas products. Since 2002, Prof. Mi YAN's team at Zhejiang University, China has carried out long-term cooperation with related enterprises, achieved key breakthroughs, and invented a series of new SMCs with enhanced performance and low loss. The team has also achieved large-scale production and wide application of the new SMCs, pushing the domestic industry into a world-leading position. Their main inventions and innovations include:

1 SMCs with core-shell structure containing multiple soft magnetic phases and new insulation coating technologies

Reduction of the loss, mainly eddy current loss of the SMCs, is a worldwide issue. Traditional phosphate coatings tend to crack during compaction and decompose during annealing (Fig. 1a), leading to a deterioration in insulation effectiveness. The team at Zhejiang University came up with an original idea of generating soft magnetic shells with large electrical resistivity surrounding the magnetic powders. They systematically investigated the kinetics and dynamics during chemical heat treatments for Fe-based alloys and obtained insights on the influences of the gas partial pressure, annealing temperature, and reaction time on the morphology and thickness of shells as well as on the interfacial structures. Based on deep understanding of the relationship between the phase composition of the shells and the performance of the SMCs, the team achieved controllable growth of the shells successfully and invented a series of novel SMCs containing Fe-based magnetic alloy cores and Fe_4N/Fe_3O_4 or Fe_3O_4 shells with high electrical resistivity (Fig. 1b). The *in-situ* grown shells exhibit significantly increased electrical resistivity compared to the alloy powders, and effectively suppress the eddy current loss. The shells also possess satisfactory soft magnetic properties to reduce magnetic dilution. Furthermore, the *in-situ* growth gives rise to excellent adhesion of the shells for maintained stability during compaction and annealing. Such core-shell technique can be applied to all the Fe SMCs and most crystalline Fe-contained alloy SMCs, giving rise to simultaneous high magnetic performance and eliminating inter-particle eddy current loss.

The team has also investigated the growth mechanisms of other insulation methods and studied

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the microstructure of different coating layers. New coating methods such as heterogeneous nucleation and sol-gel have also been applied for insulation coatings. For example, uniform Al_2O_3 and MgO coatings have been fabricated which exhibit excellent adhesion with the magnetic powders, and their thickness has been reduced to just dozens of nanometers. Consequently, the eddy current loss can be eliminated effectively with the minimum introduction of magnetic dilution. Not only can such new technologies be used to produce amorphous and nanocrystalline SMCs as well as Co-based SMCs where it may be difficult to form the core-shell structure, they can also be used, if necessary, to further reduce the loss for the core-shell composites.

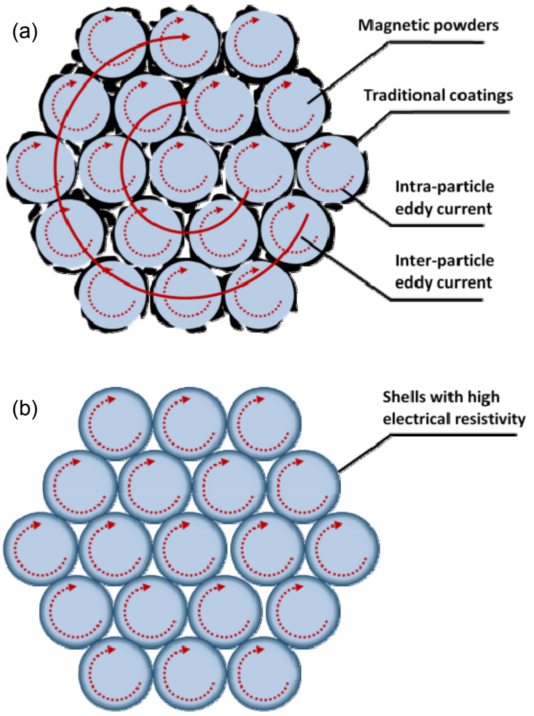

Fig. 1 Core-shell structure with multiple soft magnetic phases significantly reducing the eddy current loss
(a) Traditional insulation coating; (b) Core-shell structure

2 New magnetic alloy systems with enhanced properties

The team has conducted long-term investigation on the composition, microstructure, and magnetic properties of Fe-based alloys and mastered understanding of the roles of different alloy elements in the evolution of the microstructure during condensation. New crystalline magnetic alloys such as Fe-Si-M and Fe-Ni-M (M is Mo, Ni, Al, or Co) with high saturation magnetization, excellent DC-bias properties and stability have been designed and produced. The introduction of alloy elements is also beneficial to reduce the intra-particle eddy current.

The team has also carried out extensive investigations on the composition, heat treatment, and magnetic properties of nanocrystalline alloys. Nanocryallites were fabricated via melt spinning followed by thermal crystallization. Based on the deep understanding of the influences of elemental substitution and thermal crystallization on crystal size, volume, and stability, new nanocrystalline alloys, such as Fe-Cu-Nb-Ti-Si-B and Fe-Ni-Al-Si-B, have been invented with superior magnetic performance.

3 Systematic invention and integration of key production technologies

To solve the thermal stability issue of traditional epoxy resins, the team has invented new organic-inorganic hybrid binders via *in-situ* growth of SiO_2 nanoparticles in the epoxy-modified silicone resin (ESR). Because of the formation of chemical bondings between the nano-SiO_2 and the ESR, significantly enhanced mechanical properties, thermal stability, and electrical resistance can be obtained. The team has also investigated the binding properties of SiO_2, Al_2O_3, B_2O_3, and glass with low melting point, and developed novel inorganic-organic hybrid binding technology. This allows heat treatment at 700 °C of the SMCs, which is beneficial for complete stress relaxation and reduced coercivity.

In terms of powder production, the team has innovatively improved the design of the key component of the atomization furnace and invented BN-based refractory material used as the nozzle of the non-vacuum atomization furnace. This guaranteed stable production of Fe-Si-M and Fe-Ni-M powders and elongated the lifetime of the nozzle by 60%. Based on simulations of the heat distribution of the cooling system for metal spinning, the team designed new cooling cycles and improved the amorphorization of the alloy ribbons. The team has also invented pre-heat treatment to improve the brittleness of the ribbons, and improved high energy ball milling conditions to produce powders with desired shape. These inventions and innovations have been integrated, and complete production lines involving powder production, insulation coating, binding, compaction, and heat treatment have been built to produce SMCs with high performance and low loss.

3 National Award for Technological Invention of China (2nd prize)

Such inventions and technological advances have reached a world-leading level and have been applied in large scale production in the participating enterprises, making important contributions to areas including new-energy vehicles, high-speed trains, computers, and motors. The inventions and innovations described above have won Prof. YAN's team the National Award for Technological Invention of China (2nd prize) in 2016. It is worth mentioning that this is the second National Award won by the team. In 2013, their contribution on the "Grain boundary reconstruction of NbFeB and key technologies to produce magnets with low cost and high performance" has also been awarded the National Award for Technological Invention of China (2nd prize).

Introducing editorial board member:

Prof. Mi YAN joined Zhejiang University, China in 1991 as a postdoctoral research fellow. During 1997 to 1999 he worked at Oxford University as a visiting research fellow, and at Brunel University as a research fellow. He is now a professor of the School of Materials Science and Engineering at Zhejiang University, the head of a research group of over 40. He is the Vice Director of the Magnetic Materials and Devices Branch of China Electronic Components Association, and the Director of Foundry Association of Zhejiang Province, China. He is also a member of the Editorial Board of *Journal of Zhejiang University-SCIENCE A (Applied Physics & Engineering)* and *SPIN*.

Prof. YAN received the State Technological Innovation Award twice (2nd prize/1st author in 2013 and 2016), the Zhejiang Provincial Science and Technology Progress Award five times (1st prize/1st author in 2006, 2009, and 2014, and 2nd prize/1st author in 2003 and 2004), and the Science and Technology Award of the Ministry of Education, China (2nd prize/1st author in 2007). He published the book *Magnetism Basics and Magnetic Materials* in 2006 and it has been reprinted ten times. He has more than 350 papers and has given many invited talks at international conferences. He also established close collaboration with industry.

His research interests are in the following areas:

1. Preparation, characterization, and performance of advanced magnetic materials, including RE permanent magnets, amorphous and nanocrystalline soft magnets, nanocomposites, and magnetostrictive materials;
2. Surface treatment of metallic and/or magnetic materials, with the aim to enhance the wear or corrosion resistance of materials;
3. Advanced casting technologies.

中文概要

题　目：低功耗高性能软磁复合材料及关键制备技术

概　要：软磁复合材料是软磁金属经制粉、绝缘处理、粘结、压制和热处理而制备的磁性复合材料，广泛应用于能源、信息、交通和国防等重要领域，是国民经济和国防建设的关键基础材料。随着电力和电子装备向高频、高功率密度、节能和电磁兼容方向发展，软磁复合材料的需求量越来越大，要求也越来越高。我国软磁复合材料过去长期存在功率损耗大、磁通密度低、直流叠加性能差等严重问题，技术水平与国外差距巨大。2002 年以来，浙江大学严密教授团队与相关企业进行了长期的合作研究，原创性提出了制备多软磁相核壳结构复合材料的技术思路，创新了绝缘包覆技术，大幅消除了颗粒间涡流损耗；建立了软磁合金新体系，显著提高了复合材料软磁性能；创新和集成核心生产技术，实现了规模化生产和广泛的实际应用。

创新点：1. **发明多软磁相核壳结构复合材料和磁粉绝缘包覆新技术。**降低以涡流损耗为主的功率损耗，是软磁复合材料的世界性难题。本项目提出了在软磁粉末基体上原位生成高电阻率软磁壳层，制备多软磁相核壳结构复合材料以降低涡流损耗的新思路，发明了 Fe 基软磁合金基体与 Fe_4N/Fe_3O_4、Fe_3O_4等高电阻率软磁壳层组成的核壳结构材料。创新了磁粉绝缘包覆技术，发明了

分别适用于不同合金磁粉的非均匀形核、溶胶-凝胶和复合绝缘包覆技术。以上发明在保持高磁性能的同时，大幅度抑制了颗粒间涡流，显著降低了功率损耗。**2. 发明系列新型高性能软磁合金。**创新设计了 Fe-Si-M、Fe-Ni-M（M 为 Mo、Ni、Al 或 Co）新型晶态软磁合金和 Fe-Cu-Nb-Ti-Si-B、Fe-Ni-Al-Si-B 纳米晶/非晶软磁新合金，掌握了成分配方对合金相结构、显微组织和磁性能的作用规律及机理，发明了系列新型高性能软磁合金，制备出具有高磁通密度、高直流叠加等不同特性的高性能软磁复合材料产品。**3. 创新和集成核心生产与应用技术。**发明了新型耐高温粘结剂和有机-无机复合粘结技术，创新和改进了针对不同合金的磁粉制备技术，系统集成相关发明与关键技术，建立了低功耗高性能软磁复合材料成套生产工艺。

成　果：项目创新成果已在合作企业全面应用，为新能源汽车、高铁、计算机及国防领域做出了重要贡献，并获得了 2016 年度国家技术发明奖二等奖。值得一提的是，这已是严密教授团队获得的第二个国家技术发明奖二等奖。2013 年度，严密教授团队完成的“钕铁硼晶界组织重构及低成本高性能磁体生产关键技术”项目亦获得国家技术发明奖二等奖。

关键词：功耗；软磁；复合材料