B04.资源材料与循环利用

分会主席:何季麟、董发勤、柴立元、张廷安、冯培忠

B04-01

钛及钛合金的低成本制备技术与研究进展

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现有的以 Kroll 法为基础的钛材利用工艺存在能耗高、污染严重、生产成本等缺陷,尤其是生产成本较高已成为限制了金属钛及钛合金的大规模使用的技术瓶颈,钛及钛合金低成本清洁制备仍旧是钛工业界的研究热点和难点。淄博傅山东北大学产业技术研究院开发出以钛氧化物为原料,多级深度还原直接制备钛及钛合金的低成本清洁制备关键技术,实现了钛材低成本清洁制备。具体包括:发明了以钛氧化物为原料铝热自蔓延-喷吹深度还原低成本制备钛合金关键技术以及镁热自蔓延-钙热深度还原规模化制备钛及钛合金粉体关键技术,破解了金属热还原直接制备金属钛与钛合金的世界性难题,形成了从钛提取到钛及钛合金的低成本清洁制备关键技术突破和创新,整体技术水平世界领先。已成功制备出氧含量为 0.09%的 TiAl合金、纯度>99.69%的高纯还原钛粉以及氧含量<0.15%的 Ti6Al4V 合金粉。项目研究正在 500 吨/a 规模的还原钛粉及钛合金粉示范工程、200 吨/a 规模的高性能钛铝基合金中试放大工程,该示范工程建设将为钛及钛合金的低成本绿色制备提供工业基础,同时推动钛材的大规模利用,市场空间在万亿元以上。

关键词:还原钛粉,钛合金合金粉,钛合金,多级深度还原,低成本制备技术。

B04-02

废铝基钴-钼催化剂的高效提取及其整体利用技术研究

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稀贵金属催化剂在石油精炼过程中广泛使用,但因催化剂失活,催化剂不可避免地面临报废而成为废催化剂。全世界石油精炼催化剂的消耗量约为 70 0000 吨/年。废催化剂因含有大量有毒有害物质而被列入《国家危险废弃物名录 2016》。另一方面,废催化剂中通常含有大量有价金属元素而具有非常高的回收价值。因此,无论是从环境保护的角度还是从节约矿物资源的角度,从石油精炼废催化剂中回收有价金属元素都具有重要意义。已有研究中,废催化剂的回收大多将关注点放在稀贵金属上,对占催化剂主体的载体氧化铝的回收研究不够。此外,还存在着产品附加值低、流程复杂、回收效益低等缺点。因此,亟需开发出一套能够实现废催化剂清洁循环与整体利用的新技术,从而在实现有价元素综合利用的同时实现回收过程的经济、环保等多重目标。

本文以典型的废 Co-Mo/Al2O3 催化剂为研究对象,采用碱溶液分段浸出分离钼铝,溶剂萃取深度纯化的工艺路线制备了高附加值的氧化钼及高比表面积的活性氧化铝,建立了铝基废催化剂高值化高效循环利用新工艺体系。第一段浸出以碳酸钠为浸取剂,在优化实验条件下,钼的浸出率可达 98.2%,而铝的浸出率仅为 2.7%,实现了废催化剂中钼的选择性回收。以碳酸根型 N263 为萃取剂进行钼铝深度分离,二者分离系数可达 1828.3。第二段浸出以氢氧化钠为浸取剂,在优化条件下,氧化铝的浸出率达 98.13%,实现了载体氧化铝的高效回收。动力学实验表明,氧化铝浸出过程的反应级数为 0.99,活化能为 45.50 kJ/mol,浸出反应受界面化学反应控制。浸出渣为镍钴富集渣,成分简单,利于镍钴回收。此外,以回收的钼酸铵为钼源,采用水热法制备了平均粒径为 13 μm、晶粒尺寸为 70.67 nm 的纯相 α-MoO3。采用氧化铝浸出液作为铝源,通过水热沉淀法成功合成了平均粒径为 2.2 μm,孔径和比表面积分别是 3.8 nm 和 310.6 m2·g-1 的 γ-Al2O3。再生制备的 γ-Al2O3 性能优良,可以用作催化剂的载体。

B04-03

硬质合金材料的绿色短流程再生技术探究

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钨是重要的战略资源,世界上很多发达国家都非常重视钨废料的回收和再生利用,废钨回收已经成为钨供应的主要来源。 我国占全球 57%钨资源储量,供给全球 80%以上需求,随着我国钨消费量的逐年增加,钨矿资源作为不可再生资源也将面 临开采枯竭。因此,钨二次资源的利用显得尤为重要,熔盐具有电化学稳定性好、可提供反应温度高等优势,利用熔盐介质 电解硬质合金材料,实现钨的高效短流程循环利用。

在 NaCl-KCl、NaF-KF 等熔盐体系中,硬质合金材料可以发生电化学溶解,碳化钨中的钨以离子形式溶入熔盐,钨离子在阴极沉积的过程是一个由扩散控制的可以过程,钨离子在熔盐中的形核机理为三维逐渐形核。在 NaCl-KCl-xNa2WO4 熔盐体系中,钨酸钠的加入有效的提高了硬质合金材料阳极的溶解效率及电流效率,有效的除去了反应过程中阳极残余的碳,提高了阴极钨粉产物的纯度。在熔盐体系中对 WC- xCo 含钴硬质合金材料进行电解回收,利用双阴极法及分步电解法可以使钨、钴分离,分别获得纳米钨粉、纳米钴粉以及多孔碳。

关键词:熔盐电解,硬质合金,阳极溶解,纳米钨粉。

B04-04

Novel Low Density Thermal-Insulation Cork Composites with High Performance

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Thermal insulation materials protect normal operation of instruments by blocking heat-transfer. Especially for aerospace instruments, thermal insulations are key structures, which protect spacecrafts from being burned or overheated in aerodynamic heating environments. With the development of modern space technology [1,2], the performance requirement of thermal insulation material has gradually improved to avoid the thermal damage caused by the aerodynamic heating generating in the process of flight on the surface of spacecraft. Some new requirements, such as low density, high heat load adaptability, high mechanical strength, low-cost, short production cycle, and so on, promotes the rapid development of high performance thermal insulation materials.

This novel cork composite exhibits low coefficient of thermal conductivity, high heat resistance, ablation resistance, high suppleness, and excellent mechanical performance. It can be processed into plates and other special-shaped boards, such as U-shaped and cylinders. These features allow conventional application of cork composite as heat-insulating coating for various parts of aerospace equipments.

Table 1. Performance parameters of cork composite

Performance Parameters	Cork
Density (g/cm³)	0.40~0.45
Thickness (mm)	1.5~5.0
Tensile strength (MPa)	1.0~2.0
Elongation at break (%)	≥ 5.0
Coefficient of thermal conductivity (W/m.K)	0.08~0.10
Specific heat capacity (kJ/kg.K)	2.1~2.4
Compressive strength (MPa)	≥ 15.0
Compressive elastic modulus (MPa)	≥ 8.0
Impact strength (kJ/m^2)	≥ 1.5
Slenderness	7.5~10.0
Shore scleroscope hardness	72~78

This cork composite is produced from cork grain, modified phenolic resin adhesive, nitrile rubber, reinforced fiber and other ingredients. The modified phenolic resin affords more than 60% carbon debris at 1000 °C, and exhibits excellent properties on high temperature resistance, instantaneous ablation resistance, mechanical performance, and flame resistance. The introduction of modified phenolic resin could greatly enhance the ablation resistance of cork composite, while nitrile rubber improves the suppleness,

and reinforced fiber enhances the tensile strength and impact-resistance strength. The physical and mechanical parameters of cork composite are summarized in Table 1.

When ablated at 800 °C, the flat surface of cork composite with a little cracks indicates its excellent ablation resistance. There is no significant deformation of cork composite under long-term stress, and a rapid elastic recovery rate is observed. The water adsorption rate of cork composite is under 1% after 24 hours of swelling test. The good waterproof performance allows the application of cork composite in humid environment.

The excellent mechanical strength and suppleness of cork composite expands its application range. Besides flat products, U-shaped, cylinder, cone, as well as other special-shaped products can also be produced according to the protection requirements of different parts of high-speed flying objects in order to avoid damaging the property of material after secondary processing and assembly.

The cork composite is a novel low-density and highly efficient thermal insulation material. It possesses low thermal conductivity coefficient, high temperature resistance, ablation resistance, high slenderness, and excellent mechanical performance. In addition, it is produced by low-cost raw material and short processing cycle. Therefore, the cork composite will meets various new requirements on modern space technology for high-performance thermal insulation.

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B04-05

Processing of hierarchically porous ceramics for Environmental Applications

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Microporous ceramics such as zeolites and aluminophosphates are high surface area materials with great potential for catalysis, filtration and gas separation applications. The application criteria require that these microporous ceramics, which are produced in powder form, are assembled into mechanically strong hierarchically porous structured bodies. In this regard, binderless processing to structure microporous ceramics to hierarchically porous adsorbents without addition of binders will be discussed. The rapid thermal processing of porous ceramic powders will be presented to produce volumetrically efficient hierarchically porous structured monoliths and laminates with properties to overcome the limitations of conventional adsorbent materials specifically for decarbonization of power-plant flue gas and biogas streams. Furthermore, nanostructurization will be discussed as a versatile tool to structure porous adsorbents that outperform conventionally structured zeolites in all aspects of post-combustion decarbonization of gas streams, including CO₂ uptake capacity, high CO₂ over CH₄ and CO₂ over N₂ selectivity, rapid uptake and release kinetics and mechanical stability. The potential of these structured microporous will be discussed for gas storage in environmental applications.

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B04-06

磷石膏基矿渣水泥改性增强实验研究

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磷石膏基矿渣水泥是以磷化工业固废物磷石膏为主要原料的一种新型低能耗水硬性建筑胶凝材料。磷石膏是以磷矿和硫 酸为原料湿法生产磷肥、磷酸时排放出的固体废弃物,呈酸性,每生产1t磷酸约产生4.5-5t磷石膏。其组成除主要成分水 合硫酸钙外,还含少量硅、镁、铁、铝、磷、氟及有机质等。世界湿法磷酸年总产量约 2.6 亿 t(以 P₂O₅ 计),副产磷石膏约 1.5 亿 t, 但磷石膏总体利用率很低。磷酸是农业、工业、食品、医药等行业的重要原材料,对国民经济及社会发展具有重要 作用,随着国民经济的快速发展,磷酸产需量日益增加,磷石膏的排放量逐年大幅递增,目前我国磷石膏堆存量已过3.0亿 t。磷石膏资源化利用迫在眉睫,近些年对其综合利用研究进行了不懈努力,但总的利用率和利用水平均很低,实际应用领 域主要为制作低端石膏板、免烧砖及缓凝剂等建筑材料。由于材料配合性差、工艺简陋,导致制品的凝结时间长、强度低、 耐久性及抗渗性能差,其市场认可度低;加上此类制品的市场容量较小,磷石膏的消化量受到很大限制,总的利用率不足 20%, 离"到 2020 年, 磷石膏综合利用率达到 40%以上"的目标还很远。磷石膏大量堆存,不仅占用土地资源,还对周边环 境带来严重影响。因此,迫切需要找到能大量无害化处置和大宗资源化利用磷石膏的科学方法和技术。文章针对目前磷石膏 基矿渣水泥凝结时间长、早期强度低等缺陷进行改性探索试验研究,对原材料和掺合料化学成分、粒度分布等理化性质进行 分析,对促凝剂、增强剂及有关外加剂等进行优化选择,确定了适宜的掺合配比,并确定了工艺方案。研究结果显示,经改 性后磷石膏基矿渣水泥及其混凝土制品性能得到明显改善,凝结时间明显缩短,早期强度显著提升,胶砂初凝时间可控制在 150min 内、终凝时间可控制在 240min 内, 7d 抗压强度可达 25MPa 以上, 混凝土初凝时间可控制在 210min 内、终凝时间 可控制在 360min 内, 3d 抗压强度可达 25MPa 以上、7d 抗压强度可达 40MPa 以上。研究结果为磷石膏工业废渣在建材领域 大体量利用的产业化技术研究和工业推广应用奠定一定基础。

B04-07

The erosion-corrosion behaviors of the full-scale P110SS tubing

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The high temperature erosion-corrosion behaviors of full-scale P110SS oil tubing in the acceleration simulation working condition of the Moxi 8 gas well were investigated in the present work.

The adopted apparatus was the high temperature and high pressure erosion-corrosion full-scale test system which was independently developed by TGRI. Measurement and inspection were performed on the macro-morphology and chemical compositions by KH-7700 digital microscope and direct-reading spectrometer respectively. The micro-morphology and composition of the corrosion products were characterized by scanning electron microscopy (SEM), X-ray power diffraction (XRD) and energy-dispersive spectroscopy (EDS).

The results indicated that the BG110SS oil tubing materials suffered catastrophic corrosion; the maximum and average pitting corrosion rate of the BG110SS in the gas phase fluid were 5.29mm/a and 2.8mm/a respectively; while those in the liquid phase fluid were 11.13mm/a and 6.21mm/a respectively; A double-layer corrosion products scale, inner FeCO₃ layer and outer mixture layer mainly dominated by FeO₃ Fe₃O₄, Fe₂O₃ and FeO(OH), formed on the BG110SS.

B04-08

Preparation of palygorskite-template amorphous carbon nanotubes for the removal of dyes from aqueous solutions

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Carbon materials are research hotspots in the field of material science because of their unique structure and property. Here, a amorphous carbon nanotubes (CNTs) was synthesized by using natural nanoscale rod-like palygorskite (Pal) as hard template and glucose as carbon resource through direct impregnation and calcination method. The nanostructures of amorphous CNTs were systematically characterized by X-ray diffraction, fourier transform infrared spectroscopy, raman spectroscopy, X-ray photoelectron spectroscopy, and transmission electron microscopy techniques. Analysis of the N₂ adsorption—desorption isotherm indicated that the obtained amorphous CNTs possess a hollow structure with a large specific area (877.09 m²/g) and pore volume. The formation mechanism of amorphous CNTs is proposed based on the characterization results. The adsorption test of congo red (CR) dye was conducted to evaluate the adsorption performance of amorphous CNTs. The maximum adsorption capacity was found to be 467.97 mg/g, which is 14-fold higher than that of Pal/Carbon composite. Furthermore, the adsorption isotherm model and kinetic model were investigated. The Langmuir isotherm model and the pseudo-second-order model matched well with CR adsorption of amorphous CNTs. The approximate mechanism underlying the adsorption of CR onto amorphous CNTs was then established. The fabricated amorphous CNTs exhibit excellent adsorption performance and may thus be a candidate adsorbent for environmental remediation.

B04-09

Effect of electrolyte circulation on metal recovery of waste CPU slot

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As the core part of computers, CPU slot contains a lot of metals and has high recovery value. In this study, slurry electrolysis is the main method, and the electrolyte recycling system has been used to recover metal from the waste CPU slot under the optimum experimental condition of the early single cycle. The distribution of metal after slurry electrolysis and 14 electrolysis cycles has been studied. The experimental results show that Al, Ni and Pb mainly exist in electrolyte or anode slag, Cu, Pd and Au mainly exist in cathode powder and electrolyte after electrolysis. Pt mainly exists in anode slag, and the existence form of Ag is influenced by the concentration of chlorine ion in the system. By comparison of single electrolysis and electrolyte electrolysis, the distribution rate of single metal and total metal in the cathode powder and the recovery rate of copper powder gradually increased. The purity of copper powder and the metal content in the electrolyte decreased, and the current efficiency of the copper powder shows little obvious regularity.

Keywords: Waste CPU Slot, Slurry electrolysis, Electrolyte circulation, Metal recovery

B04-10

残余铁氧化物在尾矿微晶玻璃制备中的行为

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随着钢铁行业的迅速发展,铁尾矿资源大量产生,大量尾矿堆存不但造成矿产资源的巨大浪费,同时也给环境造成严重 的压力。对铁尾矿资源的二次回收利用已成为涉及资源及环境可持续发展的重要课题,收到国内外学者的广泛关注。

在以铁尾矿为主要原料制备矿渣微晶玻璃的过程中,残余铁氧化物的存在对微晶玻璃的析晶过程、晶相组成和微观结构产生很大影响。本文基于鞍山地区铁尾矿成分特点,以化学纯试剂作为原料,根据 $MgO-Al_2O_3-SiO_2$ 三元相图堇青石微晶玻璃组成范围设计基础玻璃配方,利用烧结法制备 $MgO-Al_2O_3-SiO_2$ 系统微晶玻璃。通过 DSC、XRD、SEM、IR、MS 等先进技术手段进行分析不同 FeO 和 Fe_2O_3 含量对微晶玻璃的种类、核化温度和晶化温度、析晶能力、微晶化的影响以及 Fe^{2+} 和 Fe^{3+} 的结构对微晶玻璃结构的作用机理,研究结果显示:

1.随着原料组份中全铁含量的增加,尾矿微晶玻璃试样成核和析晶温度降低,试样组织由玻璃相向柱状结晶再向粒状结晶 最后到细小球状晶粒转变。这是由于系统中铁氧八面体结构增加,削弱了网络结构,促进玻璃分相,为玻璃析晶提供驱动力, 为析晶提供更多的成核位,促进质点移动,导致更多晶体析出,同时析晶温度降低,热处理温度较高,导致柱状结构断裂, 使微晶玻璃形态发生变化,导致晶体数量增多,晶粒变小,结构致密。

 $2.Fe^{2+}$ 处于八面体配位结构,属于网络外体离子,主要起促进析晶作用; Fe^{3+} 一部分处于四面体配位,此时主要起增强玻璃网络连接作用,另一部分处于八面体配位结构,处于八面体配位结构时和 Fe^{2+} 作用相同。 Mg^{2+} 处于八面体配位结构, Al^{3+} 处于四面体配位结构,基于类质同相替代, Fe^{2+} 可替代 Mg^{2+} , Fe^{3+} 可替代 Al^{3+} 。因此随着铁氧化物含量的增加尾矿微晶玻璃主晶相由革青石相向尖晶石相转变。

B04-11

Primary investigation on microwave heating technology applied on metal material melting process

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Microwave heating technology applied in many fields due to its clean characteristic. However, as to be a strongly reflective material, metal can not be heated by microwave directly. This research explored the feasibility of microwave melting metal material by adopting special device and changing the original state of metal material. The study indicated that: At high temperature, metal material exerted a good microwave absorbing performance. By using SiC crucible, the melting process could be achieved by three steps: crucible heating metal, both crucible and metal heating and metal heating crucible. Fine raw materials would get increasing absorbing microwave area, so metal microwave melting technology would have widely future applications for metal recovery.

B04-12

废锂电池负极材料的绿色回收与再生研究

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锂离子电池是当前广泛应用于便携式电脑、手机、数码设备等的绿色电源,其使用寿命只有 1-3 年。随着锂离子电池产量的增加,大量废锂电池应运而生。废锂电池中含有有机物和贵重金属元素,若不及时处理会对人类造成危害,同时也会造成资源浪费。锂离子电池的负极一般是由铜箔(占负极总质量约 30%)和负极活性材料(其中 97%为石墨)组成。在充放电过程中部分的锂离子会插入到负极活性材料的介孔中,因此报废的锂离子电池负极活性材料中会聚集一定含量的锂。锂作为一种稀有金属,若不循环利用预计在 2023 年会出现短缺。可见,负极材料中含有可供二次利用的铜箔、石墨以及稀有金属锂。因此对废锂电池负极材料进行绿色回收以及循环利用是十分有意义的。

湿法冶金方法由于操作条件温和、可工业化等优点被广泛应用于回收领域。本文以废锂电池负极材料为原料,选取三氟乙酸作为溶剂,研究了三氟乙酸对负极活性材料与铜箔的分离效果;考察了三氟乙酸浓度、固液比(负极材料质量与三氟乙酸体积)、反应温度以及反应时间等条件对废锂电池负极材料中锂离子浸出效果的影响。研究结果表明三氟乙酸能够很好地使负极活性材料从铜箔分离,铜箔的回收率可达 98%。最优浸出条件为:三氟乙酸浓度 15%、固液比 60 g/L、反应温度 40℃、反应时间 30 min,此时浸出液中锂离子和铜离子的浸出百分含量(占负极材料总质量的比例)分别为 1.08%和 0.82%。浸出液经真空抽滤,可得黑色滤渣;通过控制适当的条件向过滤液中滴加氢氧化钠,经过滤后铜离子的去除率可达 96.8%;然后向滤液中以一定的化学计量比滴加饱和碳酸钠可得白色沉淀。XRD 和 SEM 分析可得黑色滤渣为高纯度的层状石墨;白色沉淀为碳酸锂,其产率为 95.6%。三氟乙酸溶剂可通过蒸馏来回收以循环使用。该回收过程工艺简单,且不产生二次污染,所回收的铜箔、石墨和碳酸锂纯度高,可直接二次利用。

B04-13

利用污泥与沸石、粉煤灰制备辅助胶凝材料

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以污泥建材资源化为目标,实验研究了污泥在干化阶段分别加入沸石、粉煤灰对干化速率的影响,并将调质污泥煅烧作火山灰质混合材料制备辅助胶凝材料。

沸石掺量为 10%、14%、18%、22%、26%的调质污泥,在 800℃、100min 得到煅烧调质污泥灰。实验结果表明,沸石掺量越多污泥干化速率越快,沸石掺量为 26%时,污泥含水率为 22%左右;沸石掺量为 18%的调质污泥制备的辅助胶凝材

料活性最佳;同时,对沸石掺量 18%的调质污泥进行了一组对比实验,实验的变量为煅烧时间,在煅烧温度 800℃,将煅烧时间设置为 80min、100min 和 120min,结果表明,在 800℃煅烧 100min 的 18%沸石掺量的混合污泥活性最好。XRD 和 SEM 测试结果表明,调质污泥中斜发沸石和石英的峰值,相对于未烧的沸石有所降低,说明在煅烧的过程中斜发沸石发生了分解,有利于调质污泥活性的提高。

粉煤灰掺量为 10%、15%、20%、25%、30%的调质污泥,在 800℃、100min 得到调质污泥灰。实验结果表明,粉煤灰掺量为 30%时,污泥含水率为 26%左右,对后续的处理和利用有利,粉煤灰掺量为 25%时制备的辅助胶凝材料活性最好。根据 XRD 和 SEM 分析可知,煅烧粉煤灰和煅烧调质污泥的矿物成分相同,均为莫来石和石英,煅烧调质污泥的莫来石的衍射峰减弱,无定型相增多,这对于调质污泥的活性较为有利。

B04-14

熔分赤泥制备无机纤维的实验研究

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以熔分后赤泥为主要原料,配加一定比例的石英沙和轻烧白云石,采用高温熔化调质-离心甩丝工艺制备无机纤维,并探究了原料配比对纤维性能的影响。结果表明,当赤泥配比在 50%~85%范围时,随着赤泥掺量的增加,纤维含水率变化幅度不大,均低于 0.5%;纤维直径和渣球含量均随着赤泥掺量的增加逐渐增大,其中纤维直径在 5.5~8.5um 的范围内波动,渣球含量由 0%增大到 11.7%,变化程度较大。综合对比纤维理化性能,原料最佳配比为赤泥渣 70%,石英砂 21%,白云石 9%,最优工艺参数为:渣温度 1599℃,离心机转速 5800r/min,离心机风环转速 2800r/min,流股流量为 14kg/min,所得纤维性能较好,质量水平达到商用建筑用外墙保温纤维水平。

B04-15

Recovery of metals from WPCBs with ionic liquid [MIm]HSO4 by slurry electrolysis

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WPCBs contain not only harmful substances, but also valuable metals, which has enormous resource recovery value. In this study, a typical ionic liquid [MIm]HSO₄ was applied to replace H₂SO₄ in a slurry electrolytic system in order to recycle metals from WPCBs, and the effects of ionic liquid [MIm]HSO₄ with different substitution rates on the leaching rate of metals, current efficiency, purity, particle size, and metal powders microstructure were investigated and discussed in detail. The results showed that the ionic liquid [MIm]HSO₄ had a certain effect on the leaching rate of metals in slurry electrolysis system, which improving the leaching rates of Cu, Zn, Al and total metals. When more H₂SO₄ was replaced by [MIm]HSO₄, current efficiency and purity of metal powders first increased and then decreased. Besides, a smaller particle size of copper powders can be obtained while adding ionic liquid [MIm]HSO₄. XRD results showed that copper was the main phase, and ionic liquid [MIm]HSO₄ had little effect on the phase composition of the metal powders. SEM and EDS results also showed that the purity of copper powder was very high and the metallic copper powder structure was dendrite structure.

B04-16

石墨烯/硫化镉高催化性能复合材料的制备及第一性原理法分析

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采用 Hmmers 法制备氧化石墨(GO)作为前驱体。通过一步溶剂热法将氧化石墨还原,然后与硫化镉结合制备了氧化还原石墨烯(RGO)/CdS 复合材料。在可见光下测试了复合材料对甲基橙的催化降解活性。结果表明,在不同 RGO: CdS 质量比的样品中,RGO/CdS-8 样品表现出优异的催化性能,降解甲基橙的降解率为 91.73%。通过第一性原理法分析发现,复合材料中添加的石墨烯还影响了 CdS 晶体的带隙及其电荷分布,对光子产生载流子的分离起到了积极的作用。此外,作

为良好的转移介质, RGO 可以帮助电子转移和高速移动。

B04-17

用于模拟高放废物固化硼硅酸盐基玻璃结构、析晶动力学和化学稳定性的研究

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通过傅立叶转换红外光谱,差示扫描量热法和产品一致性测试试验研究了 CeO_2 的掺杂对硼硅酸盐玻璃结构,析晶动力学,玻璃转化动力学和化学稳定性的影响。研究结果表明, CeO_2 的掺杂导致了 $[BO_3]$ 基团中非桥氧键的增加和 B-O 键的减少;掺杂 $5wt\%CeO_2$ 的样品(Ce5)拥有最大的析晶活化能 $321kJ\cdot mol^{-1}$,掺杂 $15wt\%CeO_2$ 的样品(Ce15)拥有最小的玻璃转变活化能 $197kJ\cdot mol^{-1}$; 所用样品的阿弗拉米指数 n 为 $1.0\sim1.5$ 。另外,在整个浸出试验过程中,Ce15 样品中 B 和 Si 的标准化 浸出率在 10^{-3} ($g\cdot m^{-2}\cdot d^{-1}$)数量级,为所有样品中最低。

B04-18

粉煤灰改性硫正极的制备及其高安全、高性能锂-硫电池研究

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在现有二次电池体系中,锂-硫电池因其理论能量密度高、成本低、污染小等优点,被认为是十分有潜力的下一代高能量电池体系。然而,由于活性物质硫的电子、离子传导性差,充放电过程中较大的体积变化,以及多硫化物的穿梭效应等问题,导致活性物质的利用率低、电池的循环寿命短,从而不利于其实际应用。通过正极改性能够解决上述问题,特别是针对多硫的穿梭效应,近年来已有大量研究报道^[1,2]。然而,大多数的方法通常涉及繁琐、复杂和昂贵的制备工艺,不利于其实际商业应用。粉煤灰是燃煤电厂排出的主要固体废物,若随意排放到环境中其有害的痕量重金属元素会对人类和其他生物造成危害。因此,研究粉煤灰的循环再利用具有重要的意义^[3]。而研究表明,粉煤灰中所含金属氧化物和磷化物可能存在抑制多硫化物穿梭效应的作用。本研究采用简单的球磨工艺直接制备了粉煤灰改性的硫正极材料,并应用于锂-硫电池。结果表明:10%粉煤灰添加的锂-硫电池在 0.2C 下进行充放电,其初始比容量达到 936mAh g⁻¹,循环 400 圈后电池的比容量还能保持在约 600mAh g⁻¹,而未添加粉煤灰的电池在 0.2C 下充放电 100 圈后其比容量降低至约 255mAh g⁻¹;更为重要的是,粉煤灰的添加能显著降低正极的可燃性,其单位质量燃烧时间较未添加粉煤灰的电池(518.08s g⁻¹)缩短了近一半(259.49s g⁻¹)。该工作由自然科学基金(51503026)和中央高校基本科研业务费(ZYGX2014J090;YJ201821)资助。

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B04-19

炭质材料的高效与增值利用

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针对石墨与生物质资源两种优势炭质资源,本文围绕石墨与生物质材料基本材料属性,概括了作者十多年来从事石墨与生物质材料的高效及增值利用的典型案例,结合多种加工方法提出了一系列石墨与生物质材料表面改性、组分调控、结构修饰、功能复合等技术,分别在热、电、电化学储能等领域,基于炭质资源集成的高热传导特性、电磁增强、高性能储能器件,突破了原有材料与器件的性能水平,实现了石墨与生物质资源高效与增值利用。

B04-20

利用调质熔融高炉渣制备无氟工业微晶玻璃的研究

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以($Fe_2O_3+TiO_2+ZrO_2$)作为复合晶核剂,利用石英砂对熔融高炉渣进行调质,调质后的高炉熔渣经过成型、退火和热处理后得到无氟微晶玻璃材料,其中高炉熔渣的质量分数达到 78 wt. %。运用高温差示扫描量热法(DSC)对调质后高炉熔渣的热学性质进行分析,利用 X 射线衍射(XRD)对热处理后的微晶玻璃材料进行物相分析,并结合 FE-SEM 对其微观形貌进行观察。实验结果表明:调质后的高炉熔渣析晶倾向明显,经过热处理后可以得到晶化程度较高的微晶玻璃材料。微晶玻璃物相分析表明辉石相和钙镁黄长石相同时存在,但随着熔融高炉渣比例的增加,微晶玻璃中的主晶相由辉石相逐渐转变为钙镁黄长石相。热处理过程中晶化温度由 933 \mathbb{C} 上升至 955 \mathbb{C} ,微晶玻璃中的晶相组成没有发生明显的变化,但密度测试结果表明微晶玻璃的致密化程度更高。微晶玻璃内部呈现出典型的层状及短柱状晶体交错生长的微观形貌,玻璃相与微晶相之间能够较好地相互咬合。力学性能测试表明,调质后高炉熔渣在 955 \mathbb{C} 条件下晶化 3 小时后,密度能够达到 2.91 \mathbb{C} \mathbb

B04-21

熔盐法处理废旧纺织纤维制备电容碳材料

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废旧纺织纤维的回收与利用是当前社会面临的重要课题。将废旧纺织纤维作为重要资源加以利用,既可以减少环境污染,同时也可以节约资源,是符合我国实现可持续发展和低碳经济的重要战略。在本文中,以废旧棉布和羊毛为研究对象,通过在熔盐中一步炭化活化,将其制备成电容性碳材料。研究了反应温度,废旧纺织材料、NaNO₃和 MS 的质量比例,反应时间对制备炭材料的结构、表面成分、比表面积,分析了不同条件下活性炭的电容性能,得出熔盐法所得炭材料的构效关系。实验发现,在氮气保护气氛中,温度为 700℃,废旧棉布、硝酸钠以及混合熔盐的质量比例为 1:1:10、时间为 60min,所得熔盐法制备的棉布基活性炭为不规则的颗粒状,内部含有大量的多孔结构,表面有丰富官能团结构,晶型为无定形炭,BET 比表面积为 1304.5m²/g,孔容积为 2.42cm³/g。 其质量比电容为 200.00F/g。能量密度为 19.37 Wh/Kg,功率密度为 450 W/Kg。1500 次循环充放电后,电容保持率为 90%。对于羊毛基制备的碳材料,可以制备成含氮的多孔碳材料。同时,研究表明经过熔盐碳化得到的蜂窝状羊毛纤维衍生碳(WFC-MSC)具有 787.079 m²/g 的比表面积和质量分数为 2.6%的氮含量。使用这种氮掺杂碳作为超级电容器电极材料,在 0.25A/g 电流密度下具有 318.2F/g 的高比电容以及良好的倍率性能。在 5A/g 的电流密度下经过 5000 次循环,比容量仍有 210F/g。此外,WFC-MSC 的对称超级电容器在中性 Na₂SO₄ 电解液和 1.8V 的宽电压范围中具有 20.2Wh/kg 的高能量密度,202W/kg 的功率密度。通过针对于废旧纺织纤维的熔盐处理技术研究,从而实现废旧纺织品转化为可用于高附加值能源材料。

B04-22

电解铝固体废物中氟化物的化学稳定性研究

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电解铝生产过程产生的固体废物包括铝灰、阳极碳渣、大修渣、废阴极块、抬包清理料,是回收价值较高的二次资源。这些固体废物中含有一定浓度的氟化物(主要是氟化钠、冰晶石、氟化铝),其中氟化钠是有害氟化物,容易随雨水渗入土壤和水体导致高氟水和高氟土壤。因此电解铝固体废物被政府列为危险废物,要求企业必须妥善处置、无害化处理或资源化利用。采用粉末料高温焙烧的方法实验研究了氟化物的化学稳定性,实验条件分为氟化物单独焙烧、添加石灰石焙烧、在氧化铝或二氧化硅存在下焙烧几种情况,结果表明:在空气中、700-950℃条件下,氟化物的稳定性顺序是氟化钠 > 冰晶石 > 氟化铝,几种实验条件下规律性一致;添加石灰石可以明显提高氟化物的化学反应活性,石灰石与氟化铝、冰晶石、氟化钠发生化学反应分别生成了氟化钙+铝酸钙、氟化钙+氟铝酸钙、氟化钙+碳酸钠;二氧化硅存在的条件下可以提高氟化物的损失率,氟化铝、冰晶石和氟化钠的损失率分别从 11.48%、2.9%和 0.9%提高到 67.15%、15.32%和 3.32%;氧化铝是否存在对

几种氟化物的反应影响不大;二氧化硅存在下添加石灰石可以进一步提高氟化钠的反应活性,损失率从11.3%提高到24.88%,反应产物不再是氟化钙+碳酸钠,而是氟化钙+氟硅酸钙;但二氧化硅是否存在对氟化铝、冰晶石与石灰石的反应影响不大,损失率和反应产物基本不变。计算了各化学反应的自由能 \triangle G值,氟化钠反应的 \triangle G较大,氟化铝反应的 \triangle G较小,冰晶石反应的 \triangle G介于二者之间,其规律性与实验结果吻合。

B04-23

废弃橡胶胶粉化再生利用研究

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采用回收废胶粉制备可应用的橡胶制品是推广废弃橡胶胶粉循环利用的基础与动力。为此,立足于废弃橡胶胶粉化再生利用技术开发与工程化,分别针对普通橡胶制品和功能橡胶制品,建立了机械力化学改性胶粉及并用化工艺、微波辅助铁氧体表面修饰与废弃橡胶半脱硫活化制备电磁功能橡胶工艺。研究表明:1)胶粉经双辊开炼或螺杆挤出机械力化学改性,破坏了胶粉交联结构,伴随之活性自由基产生;促进剂和改性剂等助剂的加入,调控了机械力化学改性过程中交联网络活化过程,提高机械力化学改性活化效果,降低操作温度,并使得并用硫化胶中胶粉与橡胶界面相容性大大改善,并用胶的力学性能得到提升。2)利用废弃橡胶干法粉碎所得到的胶粉为原料,加入铁氧体超细粉体,辅以微波作用实现橡胶高分子对铁氧体表面原位修饰与废弃橡胶半脱硫活化,同时解决无机纳米粉体在基体中的相容性与再生橡胶制品脱硫回收的问题,并赋予所制备磁性功能橡胶优良的磁性能,建立了废弃橡胶复合电磁功能化再生的新途径。

B04-24

含磷酸锆钠(NZP)晶相的玻璃陶瓷固化体的合成及结构和化学稳定性能的研究

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用传统的玻璃反应烧结法合成了含 $NaZr_2(PO_4)_3$ 晶相的玻璃陶瓷固化体。采用 XRD、SEM、FTIR、Raman 和 PCT 等方法研究了 ZrO_2 取代 Na_2O 对 $20Na_2O-28.8Fe_2O_3-8B_2O_3-43.2P_2O_5$ 玻璃固化体的析晶性能及析晶后结构和化学稳定性的影响。结果表明, $NaZr_2(PO_4)_3$ 是所有样品的主晶相。当 ZrO_2 的取代量超过 12mol%时(包括 12mol%), $NaZr_2(PO_4)_3$ 的含量逐渐减少且磷酸铁 $FePO_4$ 从玻璃中析出。随着 ZrO_2 取代量的增加, ZrP_2O_7 析出量增加。红外和拉曼显示玻璃陶瓷的结构由大量的正磷酸基团,焦磷酸基团,硼氧四面体单元和少量的偏磷酸基团组成。随着 ZrO_2 的取代量的增加,偏磷酸基团向正磷酸基团和焦磷酸基团转化。玻璃陶瓷的粉末样品在 90°C蒸馏水中浸泡 28 天后,Zr、Fe、Na 和 P 元素的浸出率几乎没有发生变化,分别为 2.5×10^{-6} g·m²·d¹, 3.3×10^{-6} g·m²·d¹, 2.5×10^{-3} g·m²·d¹ and 7.5×10^{-4} g·m²·d¹。

B04-25

废旧线路板处理技术与装备研究进展

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废旧线路板主要由金属组分(铜、铁、铝、锡、金、银等)、有机组分(溴化环氧树脂等)和玻璃纤维组成,具有极高的资源再利用价值,其低碳全资源化清洁处理问题已经成为全球关注的热点。本文在论述线路板产业现状与回收现状的基础上,阐述废旧线路板处理技术与装备的最新进展,特别是连续式低温热解生产线的开发,实现了废旧线路板的无害化、减量化与资源化处理,契合当前"环保型社会"的发展理念。

B04-26

废旧硅钼棒的全组分回收及其硅化物基耐高温涂层的研究

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MoSi,是一种非常有潜力的高温结构材料,但目前最广泛的应用是硅钼棒,我国年产量超过五百万吨,价值数亿元,如

何综合回收利用废弃硅钼棒是本文思考的内容。基于"整体资源化回收利用"的思想,本文在前期选择性回收稀有金属 Mo 的基础上,提出以废弃硅钼棒为原料,在钼基合金表面制备耐高温抗氧化涂层,实现废弃硅钼棒的全物质再利用。结果表明:将废旧硅钼棒进行粉碎研磨,然后焙烧处理获得 MoO_3 和 SiO_2 ,接着在 850° C进行热蒸发回收得到 MoO_3 ,该回收产物具有良好的光催化性能。进而以粉碎的废弃硅钼棒粉末为原料,采用等离子喷涂工艺在 Mo 合金表面制备获得了和基体结合良好的 $160~\mu m$ 厚的硅化物涂层,初步探明具有良好的高温抗氧化性能。即通过本研究实现了废旧硅钼棒的全物质循环利用。

B04-27

A novel approach for the recycling and reusing of silicon slurry waste

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In silicon wafer manufacturing for solar cells, the hazardous sawing waste results in serious environmental problems. In this paper, a novel approach for the recycling and reusing of slurry waste is reported. The results show that slurry waste is recycled and reused completely, and composite material of Si3N4-Si2N2O-SiC is prepared. The residual gas from oxygen enrich gas production is used, which reduced cost effectively. In addition, the reaction is exothermic, which is also energy saving and ensure the production to be continuous without external heat source. The mechanism was also discussed in this paper.

This method is reported for the first time and provides many advantages. First, the slurry waste is complete reused, and the pollution of the slurry waste reduces to zero. Second, the residual gas contained N_2 and O_2 is used, which is almost zero cost. At last, the reaction in the furnace is exothermic, so the nitridation can process without external heat source. Thus, the production of the composite materials is continuous. In fact, the discussed process was brought into production for several years.

B04-28

利用油页岩渣制备 Al-MCM-41 介孔分子筛

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本文以油页岩渣为原料,采用矿物化学处理方法提取硅源和铝源,以十六烷基三甲基溴化铵(CTAB)为模板剂,采用一步水热合成法制备 Al-MCM-41 介孔分子筛。通过 XRD、TEM、N 2 吸附-脱附等技术研究了合成条件对 Al-MCM-41 介孔分子筛的影响。结果表明,在硅铝比为 73:1、pH 值为 10、晶化温度为 65℃时,制备的 Al-MCM-41 介孔分子筛具有有序的六方孔道结构和较大的比表面积。

B04-29

煤气化高温烟气净化用 Fe-Al 基多孔材料的制备及性能研究

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Fe-Al 金属间化合物具有优良的力学性能、优异的高温抗氧化和抗硫化性能等,由于其室温脆性和高温强度不足的缺陷阻碍了它作为结构材料的实用化进程。但是将 Fe-Al 的优异性能与多孔材料的功能特点结合起来制备出多孔材料,从而解决传统多孔金属材料抗氧化以及抗硫化性能差的缺点,为煤气化行业极端条件高温烟气过滤提供选择。本文以 Fe、Al 粉末为原料,采用热爆合成法快速制备了 Fe: Al 摩尔配比分别为 60:40、55:45、和 50:50 的三种 Fe-Al 基高孔隙多孔材料,对其燃烧合成特征、相组成、微观形貌、孔结构和抗氧化及抗硫化性能等进行了分析。实验结果表明:随着 Al 含量的增加,Fe-Al 体系的热爆起始温度随之减小,而燃烧温度表现为升高的趋势,最高燃烧温度达 1134 ℃。经 1000 ℃ 均匀化烧结后,均得到单一 FeAl 相。Fe-Al 多孔材料的孔隙率为 54.2%-59.8%, 平均孔径为 26.1-31.89 μm。多孔材料主要由连续的颗粒骨架、骨架之间的大孔隙和骨架内部的小孔隙构成。Fe-Al 材料表现出良好的高温抗氧化及抗硫化性能,且随着 Al 含量的增加,材料的氧化和硫化速率显著降低。

B04-30

造孔剂法制备高孔隙率 TiAl3 金属间化合物及其孔结构研究

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通过 Ti/TiH₂和 Al 粉热爆合成多孔的 TiAl₃金属间化合物,并在此基础上添加 NaCl 或者尿素作为短暂的造孔剂材料制备出具有高孔隙率分等级的多孔材料。本文通过混料-球磨-压制-烧结工艺,并采用层层压制工艺获得了具有梯度对称结构的复杂孔隙,结果表明生坯中的造孔剂可以在烧结前被完全移除,样品在 650-670℃之间会被点燃并且样品的实际温度会在几十秒内迅速升高至 800-1200℃,表明发生了明显而剧烈地热爆反应,样品均发生了不同程度的膨胀现象。烧结坯的孔隙率可高达 70%以上,并随造孔剂含量的增加显著提高,多孔的 TiAl₃金属间化合物表现出三种孔结构:复制造孔剂颗粒的大孔隙像 NaCl (200-500μm)和尿素(700μm),热爆反应过程中产生的小孔隙和在高温阶段析出的微小孔隙,不同的孔结构均发挥着不同的作用。此外,设计出了具有三层或五层的梯度对称的复杂孔结构,使得该多孔材料能够有效地应用于特殊的过滤装置、分离材料及催化剂等领域,作为可循环使用的新型多孔材料将得到更多学者的关注和研究。最后分析了其反应机理及孔隙形成机制。

墙展

B04-P01

用于合成 MFI 型分子筛渗透汽化膜的二氧化硅支撑体的制备

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国药和科密欧公司生产的 EDTA 的颗粒均为微米级,有较多的大颗粒。在玛瑙研钵中研磨 30 min 后发现,国药的 EDTA 已成为较为均匀的细小颗粒,而科密欧的 EDTA 仍然有数十至上百微米的颗粒,且压片后制成的支撑体,肉眼即可看到有明显的大洞。使用研磨后的国药 EDTA 压制的支撑体表面大洞缺陷的尺寸和数目有所减少。为了进一步减少缺陷的数量,将研磨后的 EDTA 分散于去离子水中,搅拌均匀后沉降 1 h,用滴管取出上层溶液后烘干。SEM 观察发现,沉降分离后得到的 EDTA 粒子为纳米级,仅有非常少的微米级的颗粒。压片后制备的支撑体表面缺陷数量大大减少,涂覆晶种后晶种层连续、均一。以该二氧化硅支撑体制备的膜进行渗透汽化测试,其分离因子为 5。在此基础上,对合成时间进行调变后发现,合成时间减少至 4h 时,制备的膜致密性更好,其分离因子提高至 14,表明其对乙醇具有较高的透过选择性。SEM 表征结果可以看到,膜表面平整均一,膜的厚度约为 3 μm。

B04-P02

碱金属氯化物熔融盐中钛离子平衡行为

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由于不同价态的钛离子在电解质中发生歧化反应,造成电流空耗,进而影响了金属钛提取与提纯的电流效率。因此,不同价态钛离子与金属钛平衡的研究对于金属钛提取与提纯过程研究具有十分重要的意义。本文的目的是系统研究在碱金属氯化物熔盐中阳离子半径对钛离子平衡行为的影响。

利用三电极体系,分别在熔融 LiCl,LiCl-KCl,NaCl-CsCl 和 CsCl 中 1023 K 条件下对 TiCl₃ 在其中的电化学行为进行研究。研究表明,在上述碱金属氯化物熔体中,Ti(III)到 Ti(IV)的阳极过程是一个简单的可逆过程。在 CsCl 中,Ti(III)可以一步直接还原成 Ti,而在 LiCl,LiCl-KCl 和 NaCl-CsCl 中出现 Ti 之前会出现 Ti(II)。通过考察熔融 LiCl 二元混合物中的歧化反应($3\text{Ti}^{2+}=2\text{Ti}^{3+}+\text{Ti}$),平衡常数 K_c ,研究在 1023 K 下碱金属阳离子半径对平衡行为的影响。结果表明随着离子极化力的降低,钛离子氧化还原对电极电位降低,平衡常数 K_c 升高。

B04-P03

Fabrication of a superhydrophobic and corrosion resistant coating on magnesium alloy via a one-step electrodeposition method $Tianxu\ Zheng^{1,2}$, $Yaobo\ Hu^{1,2}$

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A superhydrophobic coating was fabricated on the surface of magnesium alloy via a fast one-step electrodeposition method. Scanning electron microscopy, energy-dispersive X-ray spectroscopy, Fourier transform infrared spectroscopy, X-ray photoelectron spectroscopy and contact angle test were used to characterize the surfaces. Corrosion behaviour in a 3.5 wt.% NaCl solution was evaluated using potentiodynamic polarization test and EIS analysis. The results show that the substrate is covered by magnesium stearate, reaching a contact angle larger than 150°. Large fraction of air is trapped by the hierarchical structure and the trapped air will largely impede the contact of corrosive media and film, thus the corrosion resistance is enhanced

仅发表论文

B04-PO-01

热爆反应 CuAl 多孔材料的组织结构与反应机制

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以 Cu 和 Al 两种粉末为原料,采用热爆反应技术制备了 Cu-Al 多孔材料,研究了原子比(Cu:Al=2:1,1:1 和 1:2)对宏观形貌、相组成和孔结构的影响,并对 CuAl 的热爆反应机制和孔隙形成机理进行了分析。结果表明:Cu、Al 原子比对燃烧温度(T_c)有显著影响,随着 Al 含量的增加, T_c 从 957℃降低到 593℃。Cu-Al 多孔材料的开孔率为 15%-32%,密度为 3.62-4.43g/cm³。孔隙主要来自于粉末压坯颗粒之间存在的原始孔、热爆反应前先熔化的 a(Al)-CuAl₂共晶相在毛细作用下发生流动形成的孔隙和热爆反应过程中由反应扩散产生的原位孔。Cu:Al=2:1 时,Cu-Al 多孔材料只含有 Cu₉Al₄相,而 Cu、Al 比例为 1:1 和 1:2 时 CuAl₂和 CuAl 两相共存。

B04-PO-02

ITO 废靶铟资源回收研究进展

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铟是液晶显示面板中铟锡氧化物(ITO)薄膜中不可替代的元素,每年用于 ITO 中的铟占全球铟消耗量的 75%以上。用磁控溅射法制备 ITO 膜所留下的 ITO 废靶料是一种经高压高温成型的铟、锡氧化物陶瓷烧结体,它耐腐蚀耐高温,但富含稀有金属铟,其回收再生技术特别是其中的铟、锡分离复杂。近年来随着科学技术的快速发展,铟需求量不断增加,研究从废旧靶材中回收金属铟有着重要的意义。本文介绍了一些从废旧靶材中回收铟的方法。

B04-PO-03

Effects of heat treatment on the microstructure, mechanical properties, and electrical conductivity of Al-Ce hypoeutectic alloy

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Al-RE (rare earth) eutectic alloy is one of the most promising conductor materials which have relative high strength, high electrical conductivity, and superior heat resistance. In this paper, the effect of heat treatment on the microstructure, mechanical properties, and electrical conductivity of hypoeutectic Al-9.26wt. % Ce (Cerium) alloy were investigated.

According to our study, the relationship between microstructure, mechanical strength, and electrical conductivity of Al-Ce alloy have been revealed using mechanical tests, electrical conductivity tests, and microstructure characterization.

During isochronous annealing treatment at 350°C, the Ce atoms are clustered by dislocations to form G-P (Guinier-Preson) zone and then form coherent nanoprecipitates, resulting in a slight increase in microhardness and electrical conductivity compared to that of as-cast alloy. Significant spheroidization of layered eutectic Al₁₁Ce₃ intermetallics was observed when annealing temperature ups to 550°C, which resulting in an increase in electrical conductivity and slight decrease in microhardness. The isothermal annealing treatment was performed on as-cast alloy at 550°C with duration ranges from 24h to 96h. As the increase of the annealing time, the electrical conductivity increased from 49.33%IACS (International Annealed Copper Standard) in the as-cast state to 53.91%IACS, and the hardness decreased from 49.1Hv to 41.4Hv.

It was found that the eutectic spheroidization was the main cause of the increase in electrical conductivity and the decrease in microhardness.

B04-PO-04

低品位硅藻土制备高活性水泥混合材研究

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在煤矿开发和硅藻土加工中堆存了大量难以提纯的低品位硅藻土,造成了严重的资源浪费和环境污染。为了更好地开发和利用这些矿产资源,将两种不同产地的废弃低品位硅藻土在 700-900℃煅烧处理,利用 TGA-DTA、PSD、XRD、FT-IR 和BET 研究硅藻土的热活化机制,利用电导率法和抗压强度法测试煅烧产物的火山灰活性,采用 XRD 研究煅烧产物对水泥水化产物物相组成的影响,并讨论煅烧产物对水泥硬化体的强度增强效应。结果表明,煅烧处理不仅能有效去除硅藻土中的有机质,而且能将其中的粘土矿物转化成为无序的活性硅铝,可显著提高硅藻土的火山灰活性。当煅烧温度为 800℃时,两种硅藻土的煅烧产物均呈现出无序程度和比表面积耦合程度好的现象,此时火山灰活性达到最大。煅烧产物对水泥硬化体强度的增强作用主要来自于微细填料效应和火山灰效应。因此,低品位硅藻土可能是未来高品质水泥混合材的重要原料。

B04-PO-05

Optimized methods for Headspace Gas Chromatography-Mass Spectrometry (HS-GC-MS) analysis of the volatile organic compounds (VOCs) at different manufacturing stages for regeneration of recycled PET

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VOC emissions from recycled PET are recognized as one of the major causes of poor healthy condition. Much attention has been increasingly focused on VOCs produced from regenerated PET for their significance in ecological safety. These emissions may be strongly influenced by the raw materials and manufacturing techniques of the recycled PET. However, there is very little published information regarding this issue. Although there are some approaches to analyze the VOCs in the air, bottles, waste water or automobile parts, these approaches are not suitable to test VOCs in fibers and textile as a result of the trace VOCs as well as the bulkiness of fibers and textile. VOCs can usually be detected by GC-MS in the low range with relatively small sample sizes. The purpose of this study was to examine VOCs releasing from reprocessed production and check the potential problems of these productions when exposed to high temperature or other extreme conditions.

In this study, we determined and compared VOC emissions from samples after different manufacturing stages such as PET popcorn, dried PET popcorn, screw melts, virgin fibers and drawed fibers, including their species and content. To simulae the VOC emissions of samples processed with the high temperature in longer time during screw melting, we investigated VOCs from screw melts utilizing Thermo Gravimetric-Mass Spectrometry (TG-MS) with 90 min in 280 °C. We found that: (1) The optimisation of the balance procedure for volatile organic compounds was performed and the optimal balance conditions were determined to be 30 min at 120 °C; (2) Seven individual VOCs were identified: considerable VOC content changes during the manufacturing process; and the drying was tremendously helpful in reducing the VOC emissions from PET popcorn; formaldehyde, acetaldehyde and TVOC were reduced by 24%, 58% and 50%, respectively after drying; while acetaldehyde, benzene, benzaldehyde and TVOC increased

dramatically after screw melting; (3) The VOCs were released at the initial stage of heat preservation (about 5 min) and VOCs content came to the maximum which indicated the almost immediate thermal degradation in screw melting.

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Effect of initial microstructure on phase precipitation and mechanical properties during heat treatment of TC21 titanium alloy

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TC21 titanium alloy is widely used in aerospace applications due to its low density-strength ratio, high strength and high toughness. So phase precipitation and mechanical properties of TC21 titanium alloy with two different initial microstructures during heat treatment were determined. Result indicated that compared with coarse microstructure alloy, fine microstructure alloy developed finer microstructure, more unstable ω and α_2 precipitates with much smaller size and lower volume fraction, and obtained better mechanical properties during heat treatment. Crack growth behavior of TC21 titanium greatly depends on the initial microstructure under the same heat treatment condition. The fine microstructure exhibits lower crack growth resistance than the coarse microstructure.

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Investigation of VOCs from the Manufacturing Process of Recycled PET Fibers using TGA-DTA/MS Technique

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Recycling is promising to dispose the wasted Poly(ethylene terephthalate) (PET) fibers currently. However, high temperature in the recycling process may accelerate the degradation of PET and residual dyes, resulting in the volatile organic compounds (VOCs) emissions, which will do serious harm to the environment and human health. Exploring the source of VOCs is significant to remove VOCs and keep products safety.

This study was carried out to investigate the influence of high temperature on the release of four VOCs (formaldehyde, acetaldehyde, benzene and toluene) during the recycling process of PET fibers, utilizing thermogravimetric analysis coupled with mass spectrometric (TGA-DTA/MS). The atmospheres and heating conditions of esterification, polycondensation and melt spinning were simulated by TGA with various atmospheres and programmed heating processes (heating from 50 $^{\circ}$ C to 240 $^{\circ}$ C in N_2 , 280 $^{\circ}$ C in N_2 and 280 $^{\circ}$ C in air, respectively, and then maintaining for 30 min). Bis(2-hydroxyethyl) terephthalate (BHET) (esterification product) and virgin PET were used to explore the thermal degradation of PET. White PET popcorn, black PET popcorn and dyes were used to explore the effect of dyes on the release of VOCs. The results indicated that: (1) formaldehyde, acetaldehyde could be generated during the manufacturing process of PET, mainly due to the thermal oxidation; (2) the disperse dyes could degrade into formaldehyde, acetaldehyde, benzene and toluene both in N_2 and air; (3) thermal oxidation would make more contributions on the release of VOCs than nonoxidative thermal degradation.