航空高性能材料与特种成形技术

航空发动机是我国的战略性行业，是航空技术发展的关键核心，对我国国防建设及航空装备发展有重要影响。航空高性能材料制备及特种成形技术是解决航空发动机“卡脖子”问题的重要基础。

目前，高功率和高可靠性航空发动机是各国航空技术竞争的核心领域。高温高可靠性航空发动机材料面临烧结制备和性能调控的难题。通过电场、力场、微波场、温度场等多场耦合技术，为高性能原料合成、材料烧结、微观结构调控和精密成型提供了新的技术方向，有望打破现有航空材料制备技术的瓶颈，在航空高性能材料制备领域具有巨大的应用前景。

一、高性能金属粉末制备技术

金属粉末是航空关键部件增材制造用原料，本方向主要针对航空领域对粉末粒度、球形度、纯度和氧含量的特殊要求，着力开发满足应用要求的制粉技术。本方向依托自主创新设计的气雾化制粉设备，重点开展金属基复合材料、高温合金、钛合金、铝合金等粉末的成分设计与制粉工艺研究。在合金凝固、粉末演变、粉末界面形成等机理上进行突破，为制备超纯净、超细金属粉末提供理论支撑，在材料的产业化应用等方面具有一定的特色与优势。

二、多场耦合烧结及成型技术

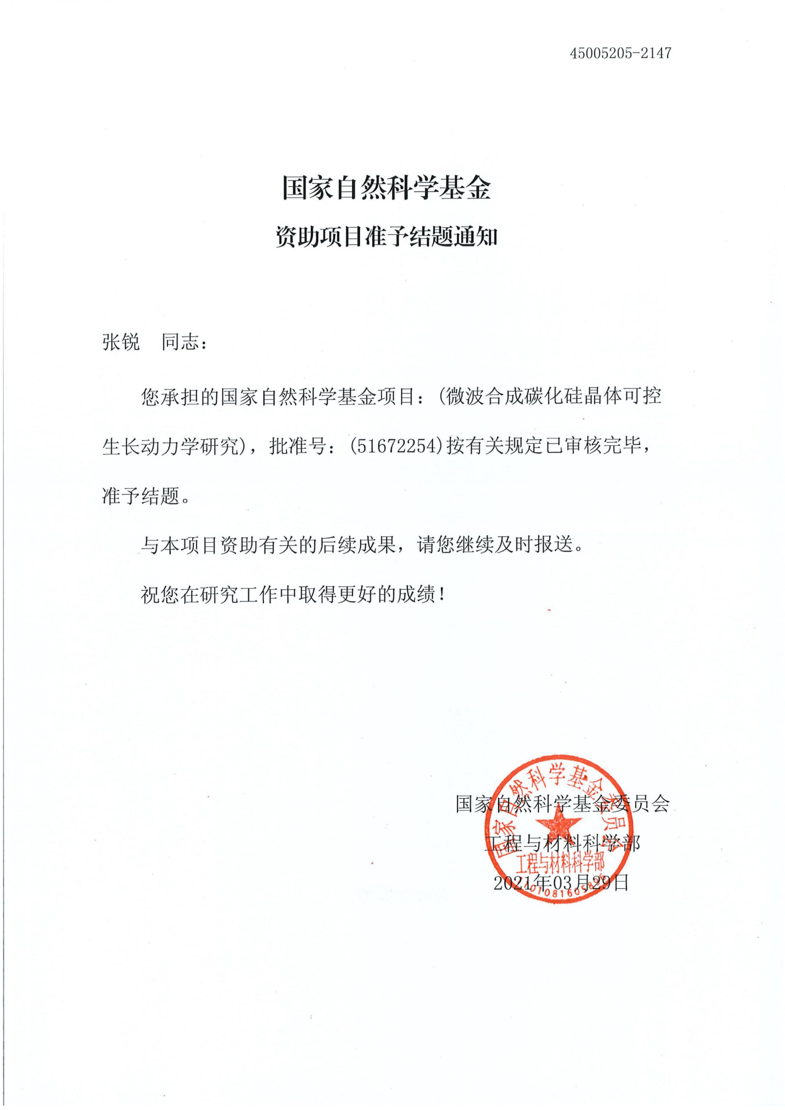
聚焦振荡力场、电场及力-热、电-热、力-电-热多场耦合下材料的烧结、变形过程、结构演化及激励机制，利用新型场辅助烧结技术（振荡烧结、振荡锻造、闪烧等）制备和成型航空用高性能金属材料、高温结构陶瓷及其复合材料，并着力解决航空航天发动机等关键部件的制备、成型和性能提升问题。先进的场辅助制备工艺是未来的发展方向，大量高性能材料将通过场辅助技术制备并广泛应用。本方向场辅助制备技术的发展将为高性能航发材料和零部件的开发和成型制造提供有力支撑。

三、微波制备技术

相比于既有的航空陶瓷材料的加热制备方式，微波制备技术具有节能、环保、改善材料性能等优点。在科技部多个项目支持下，本团队十几年来完成了从微波加热特性、微波制备机理、微波设备开发到系列制品推广应用系列成套技术的开发，已经指导工业化生产并得到了广泛的推广应用，取得了极大的项目社会效益贡献，对于航空高性能陶瓷材料的微波制造领域具有深远的影响与前沿带动作用。

本团队承担国家自然科学基金3项，航空基金3项，省部级科研项目10余项，发表相关学术论文90余篇，授权国际发明专利2项，国家发明专利20余项，获得省部级科技奖励3项；指导学生荣获大学生 “互联网+”等学科竞赛国家级奖励2项，省级奖励5项。本团队的研究成果受到北京航空材料研究院、中科院金属研究所、上海硅酸盐研究所、北京钢研高纳科技股份有限公司、清华大学、哈尔滨工业大学、西北工业大学、北京科技大学、中佛罗里达大学和印度理工学院等国内外单位广泛关注、认可和合作。

代表性科研项目



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国家自然科学基金委员会
工程与材料科学部
2021年03月29日

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根据《国家自然科学基金条例》和专家评审意见，国家自然科学基金委员会（以下简称自然科学基金委）决定批准资助您的申请项目。项目批准号：51904277，项目名称：高比重W-Ni-Fe难熔合金的振荡压力烧结机理及工艺优化研究 ，直接费用：25.00万元，项目起止年月：2020年01月至 2022年 12月，有关项目的评审意见及修改意见附后。

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附件：项目评审意见及修改意见表

国家自然科学基金委员会
工程与材料科学部
2017年8月17日

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请尽早登录科学基金网络信息系统（https://isisn.nsfc.gov.cn），获取《国家自然科学基金资助项目计划书》（以下简称计划书）并按要求填写。对于有修改意见的项目，请按修改意见及时调整计划书相关内容；如对修改意见有异议，须在计划书电子版报送截止日期前提出。**注意：请严格按照《国家自然科学基金资助项目资金管理办法》填写计划书的资金预算表，其中，劳务费、专家咨询费科目所列金额与申请书相比不得调增。**

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附件：项目评审意见及修改意见

国家自然科学基金委员会
工程与材料科学部
2016年8月17日

证书编号：ZYQR201912129

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代表性论文

Journal of Alloys and Compounds 894 (2021) 162366

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Hot oscillating pressed FGH4096 nickel-based alloy with enhanced ductility and strength

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ABSTRACT

FGH4096 Nickel-based superalloy was prepared by hot oscillating pressing (HOP) and hot pressing (HP). Compared with HPed sample, the sample prepared by HOP exhibits lightly higher yield strength and ultimate tensile strength, but greatly improved elongation. The elongation of the HOPed sample is as high as 36.9%, which is not only much higher than that of the HPed sample, but also higher than that of the sample prepared by hot isostatic pressing (HIP). The improvement in the elongation of the HOPed sample is likely due to the application of the oscillatory pressure, which enhanced inter-particle boundary sliding and/or plastic deformation during densification process.

1. Introduction

Polycrystalline nickel-based superalloys are an important class of structural materials with widespread applications due to their excellent mechanical properties [1]. The materials are usually fabricated by cast and wrought process or powder metallurgy (PM) process. Compared with the cast and wrought process, PM process is more attractive since it allows for more alloying elements and better microstructural control, so that the materials with better mechanical properties and corrosion resistance can be achieved [2–4]. The most promising sintering technique for PM superalloys is hot isostatic pressing (HIP). However, as-HIPed superalloys cannot be directly used since they usually have coarse grain structures and detrimental prior particle boundaries (PPBs) defects, which decrease the mechanical properties and limit the applications of the materials [5–12]. As-HIPed superalloys require additional hot working (such as isothermal forging, extrusion and rolling) to refine their microstructures and remove PPBs defects [13–17]. While the HIP plus hot-working route can form the materials with improved microstructures and mechanical properties, it greatly increases cost, complicates process, and prolongs processing cycle. Therefore, there is an urgent need to develop new sintering technique that can produce PM superalloys with desired properties in a simple and cost-efficient manner.

Recently, a new sintering technique - hot oscillating pressing has been developed [18]. The technique is very similar to conventional hot pressing, except that oscillatory pressure is used instead of static pressure. HOP has been applied to fabricate various materials, including ceramics, cemented carbide and refractory alloys [19–24]. The previous studies showed that the oscillatory pressure of HOP could effectively improve densification, suppress grain growth, and improve the mechanical properties. So far, HOP has not been employed to manufacture Ni-based superalloys.

In this study, Ni-based superalloy was prepared by both HP and HOP. We demonstrated that HOP can effectively inhibit the formation of PPBs. The ductility of sample prepared by HOP is much higher than that of samples prepared by HP and HIP.

2. Experimental procedures

The starting material used here is commercially available argon-atomized nickel-based superalloy powder (FGH4096) with a particle size below 53 μm. The powder is provided by Beijing CISRI-GAONA

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Flash Sintering of dense alumina ceramic discs with high hardness

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ARTICLE INFO

Keywords:
Alumina ceramics
Flash sintering
Hardness
Fracture toughness

ABSTRACT

MgO-doped-Al_2O_3 ceramic discs were fabricated by flash sintering (FS) and pressureless sintering (PS). The results showed that MgO-doped Al_2O_3 exhibited typical characteristics of flash sintering under an electric field in excess 2500 V/cm. Compared with the PS- fabricated specimen, the flash sintered specimens exhibited sub-micron grains ($\leq$760 nm) and homogeneous microstructures. The relative density of the flash sintered MgO-doped Al_2O_3 ceramics increased with current density, reaching 99.91 % when the current density increased to 7 mA/mm^2. The FS-fabricated sample exhibited higher hardness (21.02 GPa) and fracture toughness (3.46 MPa $m^{1/2}$) than PS-fabricated sample.

1. Introduction

Alumina (Al_2O_3) ceramics are among most commonly used advanced structural ceramics in industries due to high hardness and strengths, good thermal stability and chemical stability [1–4]. However, as other advanced ceramics, one of the biggest problems in manufacturing Al_2O_3 ceramics by pressureless sintering (PS) is high temperature and long time required for sintering and densification [5], which inevitably results in abnormal grain growth, and deteriorating performances of the ceramics [6].

In the past decades, aiming to short sintering time, reduce sintering temperature and improve properties of the densified ceramics, several new sintering approaches have been developed [7], such as pressure-assisted sintering including hot pressing (HP), hot oscillatory pressing (HOP) and hot isostatic pressing (HIP) [8–11], spark plasma sintering [12], microwave sintering [6], field assisted sintering techniques [13] and so on. Despite significantly positive results obtained from these new approaches, some issues, such as requiring specialized and expensive apparatus, significantly limiting the geometry of the samples produced, still exist in fabrication of these densified materials by above methods. Thus, it is attractive to explored simple and high energy efficiency methods for fabricating densified ceramics.

Since it was firstly reported by Raj and his co-workers in 2010, flash sintering (FS), as a novel sintering technology, has attracted increasing attentions due to a combination of simple process, low-cost and high energy efficient [14]. Generally, the consolidation time for flash sintering can be reduced to few seconds (less 1 min) from some hours for conventional technologies, the consolidation temperature is also decreased by hundreds of Celsius [15,16]. Until now, many ceramic materials, including ionic conductors, semiconductors and insulators, have been successfully fabricated by this novel sintering method [17–21]. Although FS has been applied in fabrications of Al_2O_3 ceramics [20,21], to the best of our knowledge, the mechanical properties of the flash sintered Al_2O_3 ceramics so far have not been reported.

In this work, as different from what have been described in previous literatures [20,21], we subjected disc-shaped MgO-doped-Al_2O_3 ceramics to flash sintering, and investigated the influence of current density on densification, hardness and fracture toughness of the FS-fabricated ceramics. As a comparison, such ceramics were also fabricated by pressureless sintering.

2. Experimental

Commercial high purity α-Al_2O_3 powder with a mean particle size of 170 nm (>99.99 %, TM-DAR, Taimei Chemical Co. Ltd., Tokyo, Japan) was used as raw material in this study. The Al_2O_3 powder doped with 0.25 wt.% MgO was prepared by adding the powder into the magnesium nitrate aqueous solution. After being sonicated for 0.5 h, the suspension was mixed by ball milling for 12 h, and then dried at 90 °C for 24 h to remove residual water. The obtained powder was sieved through a 100-mesh nylon screen, subsequently, pressed into disc-shaped ($\varphi = 10 \pm 0.1$ mm, $h = 2 \pm 0.1$ mm) green compacts with a uniaxial pressure of 200 MPa, followed by cold isostatic pressure of 200 MPa for 2 min.

Prior to the flash sintering (FS), the disc-shaped green compacts were

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Oscillatory pressure sintering of WC-Fe-Ni cemented carbides

Yang Gao[a], Ka Gao[a], Lei Fan[a], Fang Yang[a], Xiaoqin Guo[a], Rui Zhang[a,*], Linan An[b,**]

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ARTICLE INFO

Keywords:
Oscillatory pressure sintering
Densification
Grain growth
Hardness

ABSTRACT

WC-Fe-Ni cemented carbide was prepared by oscillatory pressure sintering (OPS) technique for the first time. Compared to HP, OPS can significantly improve densification and suppress grain growth of the material. The samples prepared by OPS exhibited higher hardness. The highest hardness obtained is $>$ 19 GPa, much higher than those ever reported for the similar materials. The improvements in sintering and mechanical properties are likely due to that the oscillatory pressure enhanced particle rearrangement and plastic flow of the liquid binder phase, as well as generated defects. The results suggest that OPS is a very promising technique for preparing high performance cemented carbides.

1. Introduction

Cemented carbides with high hardness, high strength, good toughness and wear resistance, have been widely used in cutting, drilling, mining, and wear resistant applications [1–3]. Among cemented carbides, WC-Co based materials show the widest applications. However, due to the environmental pollution and high cost of cobalt, it is of a great interest to replace Co by more cost-effective and less polluted metals [4,5]. It has been reported that the combinations of Fe and Ni are ideal binders alternative to Co for their low price, low pollution and good wettability with WC [6,7]. However, the applications of WC-Fe-Ni cemented carbides have not been broadly promoted yet [7], mainly due to the lack of suitable techniques that can prepared WC-Fe-Ni alloys to high density and good mechanical properties in a cost-efficiency way. A variety of sintering techniques have been explored, aiming at achieving high density and reducing grain growth. Nowadays, the pressure-assisted sintering, such as hot pressing (HP) [8], hot isostatic pressing [8,9], and spark plasma sintering [2,4], has been shown to be a promising technique for preparing WC-based materials with high density and uniform microstructures. All these techniques, which use a static pressure for enhancing plastic deformation and increasing driving force for diffusion [10], have reached the limit of reducing pores and refining grain size. Further improvements call for new processing techniques.

Recently, a novel oscillatory pressure sintering (OPS) was developed [11]. The technique is similar to conventional HP, excepting that the static pressure in HP was replaced by an oscillatory pressure. So far, the technique has been successfully used for fabrication of structural ceramics (ZrO_2, ZTA, Al_2O_3 and Si_3N_4, etc.) [11–14], as well as refractory metals [15]. The results revealed that OPS can significantly enhance the densification and suppress grain growth of the materials. The materials prepared by OPS generally exhibited better mechanical properties as compared to those prepared by conventional HP.

In this study, we report for the first time the preparation of WC-Fe-Ni alloy using OPS. We show that OPS can improve the densification and reduce grain size of the material compared to HP. We also show that the OPS prepared sample exhibits very high hardness, higher than those ever reported for the similar materials.

2. Experiments

Commercial WC, Fe and Ni powders listed in Table 1 were used as the starting materials. The received powders, in the proportion required to form 90 wt% WC, 7.5 wt% Fe and 2.5 wt% Ni, were mechanically mixed together by ball milling in a planetary mill at 120 rpm for 24 h to achieve uniform mixing [16]. The milling was carried out in argon atmosphere using a cemented carbide vial as the container, cemented carbide balls of 10 mm as the milling medium and 3 wt% alcohol as the process control agent. The resultant powder mixture were put into a graphite die of 30 mm in diameter and sintered in an OPS furnace (OPS-2020, Efield Materials Technology Co., Chengdu, China) under vacuum of 10^{-3} Pa using the following sintering schedule. First, the sample was heated to preset temperatures (1120 °C, 1160 °C, 1200 °C, 1240 °C, 1280 °C and 1320 °C, respectively) at a heating rate of 8 °C/min under a constant pressure of 30 MPa. The sample was then held at the

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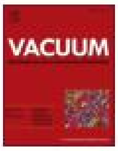

Sintering process in hot oscillatory pressure of 90W–7Ni–3Fe refractory alloy at different time

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ABSTRACT

90W–7Ni–3Fe refractory alloys were prepared by hot oscillating pressure (HOP) and hot pressuring (HP) under vacuum condition at different sintering time. Density, grain size and hardness of pure samples were measured to interpret the influence of sintering time on sintering process. Because of the increasing sintering time, the influence of sintering temperature and oscillating pressure was extended. In a shorter sintering time (about 1h), the densification rate was higher, the density increased fast, the grain growth was not obvious, and higher hardness was obtained. When the sintering time was longer (more than 1h), the densification rate was significantly reduced, and little improvement on density. At this process, the grain size was significantly increased, resulting in a decrease on hardness. However, though vacuum conditions avoid impurity elements, the measurement shows that above of HOP sample was always better than that of HP at a set value of sintering time. Whatever, these results demonstrated that HOP is a better technology to sintering refractory alloys compared with HP.

1. Introduction

W–Ni–Fe alloys are typical tungsten heavy refractory alloys used in the situations requiring high density, such as radiation shields and kinetic energy penetrators [1–5]. Recent research results show that dynamic pressure can clearly promote powder densification and improve material strength [6], then a new sintering technique [7] was proposed by Xie and An et al., hot oscillating pressure sintering technology (HOP), which is similar to traditional hot pressing (HP) sintering, the sample can be sintered within vacuum or protective atmosphere, while the static pressure imposed in HP is replaced with a dynamic oscillating pressure. This technology has been widely used in a wide range of materials from ceramics to metals [8–13], such as ZrO_2 ceramics, Al_2O_3 ceramics, ATZ ceramics, W–Ni–Fe refractory alloys, WC carbides, etc. These studies had shown that HOP can significantly reduce sintering temperature, promote densification, and inhibit grain growth by facilitating particle re-arrangement, crystal boundary slip, and pore migration.

So far, in previous report, sintering temperature and sintering pressure was the main considered in the sintering process, including HOP. In this respect, very few scholars reported sintering time. For instance, Gao et al. [12] and Wang et al. [13] fabricated WC cemented carbides by HOP and HP at different sintering temperatures. Li et al. [4,5] used 3D printing to manufacture high-performance refractory alloys at different temperatures. However, how long the sintering temperature and sintering pressure applied to the sample was determined by sintering time, then has the influence on densification, grain growth and properties. In these works, though the HOP samples were shown to be improved over those HP samples at different sintering temperature, they did not reveal the influence of sintering time during sintering, especially the effect of sintering time on densification and performance has not been discussed. Research on sintering time should be carried out.

Hence, in this paper, 90W–7Ni–3Fe refractory alloy was prepared by both HOP and HP at different range of time from 0.5 h to 2 h. Density, grain size and hardness of samples sintered were measured at different time, densification and kinetics of grain growth was obtained to

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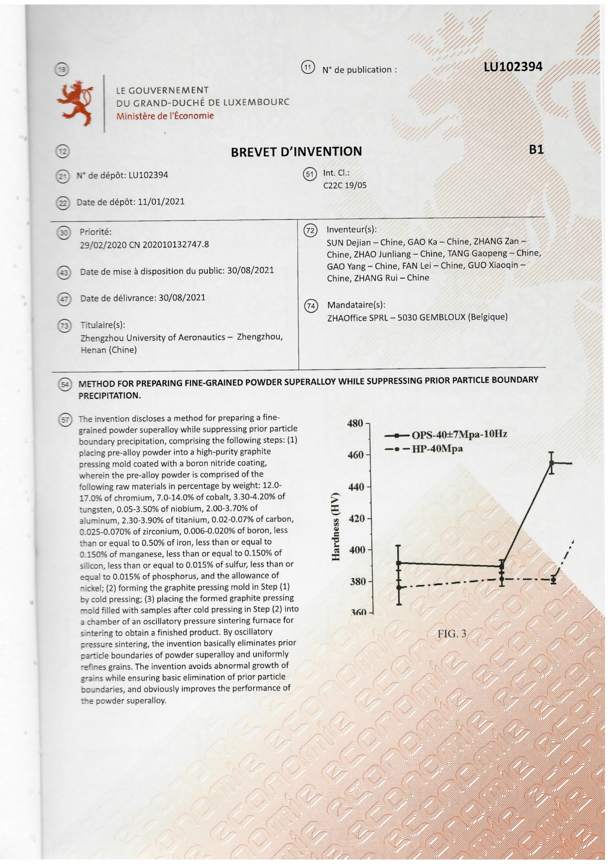

⑲ LE GOUVERNEMENT
DU GRAND-DUCHÉ DE LUXEMBOURC
Ministère de l'Économie

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⑫ **BREVET D'INVENTION** **B1**

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(54) **METHOD FOR PREPARING FINE-GRAINED POWDER SUPERALLOY WHILE SUPPRESSING PRIOR PARTICLE BOUNDARY PRECIPITATION.**

(57) The invention discloses a method for preparing a fine-grained powder superalloy while suppressing prior particle boundary precipitation, comprising the following steps: (1) placing pre-alloy powder into a high-purity graphite pressing mold coated with a boron nitride coating, wherein the pre-alloy powder is comprised of the following raw materials in percentage by weight: 12.0-17.0% of chromium, 7.0-14.0% of cobalt, 3.30-4.20% of tungsten, 0.05-3.50% of niobium, 2.00-3.70% of aluminum, 2.30-3.90% of titanium, 0.02-0.07% of carbon, 0.025-0.070% of zirconium, 0.006-0.020% of boron, less than or equal to 0.50% of iron, less than or equal to 0.150% of manganese, less than or equal to 0.150% of silicon, less than or equal to 0.015% of sulfur, less than or equal to 0.015% of phosphorus, and the allowance of nickel; (2) forming the graphite pressing mold in Step (1) by cold pressing; (3) placing the formed graphite pressing mold filled with samples after cold pressing in Step (2) into a chamber of an oscillatory pressure sintering furnace for sintering to obtain a finished product. By oscillatory pressure sintering, the invention basically eliminates prior particle boundaries of powder superalloy and uniformly refines grains. The invention avoids abnormal growth of grains while ensuring basic elimination of prior particle boundaries, and obviously improves the performance of the powder superalloy.

FIG. 3

⑲

LE GOUVERNEMENT
DU GRAND-DUCHÉ DE LUXEMBOURG
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⑫

BREVET D'INVENTION

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(54) **METHOD FOR PREPARING HIGH-DENSIFICATION TUNGSTEN-COPPER REFRACTORY ALLOY.**

(57) The present application discloses a method for preparing a high-densification tungsten-copper refractory alloy, comprising the following steps: (1) placing prefabricated powder obtained after mixing tungsten powder and copper powder into a high-purity graphite pressing mold coated with a boron nitride coating; (2) forming the graphite pressing mold in step (1) by cold pressing; (3) placing the graphite pressing mold which is filled with samples after cold pressing in step (2) into the chamber of the oscillating pressure sintering furnace for sintering to obtain the finished product, the present application adopts an oscillating pressure sintering approach, such that the tungsten-copper alloy is subjected to a multi-field coupling reaction of a heat field and a force field in the insulating graphite mold to form a copper mesh by melted and solidified then filled in gaps between tungsten particles. And a circulating pressure can drive powder to be re-arranged, liquid phase to be flowed and hole to be discharged . Finally the high-densification refractory alloy is obtained after jollification and forming. The obtained alloy basically reaches the theoretical densification. Moreover, the tungsten-copper refractory alloy prepared by the present application performs melting and solidification reactions only, and the precipitated phase is simple.

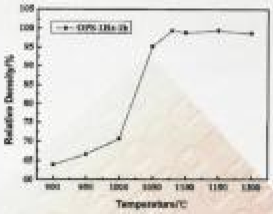

FIG. 1

证书号第3590555号

发明专利证书

发明名称：一种人造金刚石的提纯方法

发明人：张锐；樊磊；陈雷明；范冰冰；刘永奇；宋晓宇；刘军威；王铖
邵刚；王海龙；赵彪；高卡；郭晓琴；李春光

专利号：ZL 2018 1 0195584.0

专利申请日：2018年03月09日

专利权人：郑州航空工业管理学院；郑州华晶金刚石股份有限公司

地址：450000 河南省郑州市二七区大学中路2号

授权公告日：2019年11月08日　　授权公告号：CN 108190883 B

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局长
申长雨

申长雨

2019年11月08日

其他事项参见背面

证书号第4321053号

发明专利证书

发明名称：一种抑制原始颗粒边界形成的细晶粉末高温合金的制备方法

发明人：孙德建;高卡;张赞;赵峻良;唐皋朋;高阳;樊磊;郭晓琴
张锐

专利号：ZL 2020 1 0132747.8

专利申请日：2020年02月29日

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授权公告日：2021年03月26日　　授权公告号：CN 111304476 B

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局长
申长雨　申长雨

2021年03月26日

其他事项参见续页

证书号第4041072号

发明专利证书

发 明 名 称：一种高致密化钨铜难熔合金的制备方法

发　明　人：高卡；孙德建；张赞；赵峻良；唐皋朋；高阳；樊磊；郭晓琴
张锐

专　利　号：ZL 2020 1 0015880.5

专利申请日：2020年01月08日

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授权公告日：2020年10月20日　　　　授权公告号：CN 111020334 B

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2020年10月20日

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0061156

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证　　书

为表彰河南省科学技术进步奖获得者，特颁发此证书。

项目名称：陶瓷材料微波制备关键技术与成套设备

奖励等级：贰等奖

获 奖 者：张锐

河南省人民政府

2019年01月15日

证书号：2018-J-78-R01/10

为表彰在全国建筑材料行业科学研究、技术创新、成果推广、高新技术产业化等方面做出突出贡献的个人和组织，经中华人民共和国科学技术部批准，设立中国建筑材料联合会·中国硅酸盐学会建筑材料科学技术奖（国科奖社字0055号）。

建筑材料科学技术奖

证　书

项目名称：陶瓷微波制备关键技术及在异型制品和氧化物粉体的应用开发

奖励等级：一等（技术发明类）

获 奖 者：张锐

证书编号：2019-F-1-02-R01

中国建筑材料联合会　中国硅酸盐学会

二〇二〇年四月

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二〇一七年五月

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奖励等级：　　壹　等

公布文号：豫教科技【2017】62 号

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