

# 基于弥散强化钨基面向等离子体材料研究进展

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**摘要:** 重点综述了国内外关于氧化物或碳化物作为强化相的钨基面向等离子体材料的力学性能、氢滞留特性以及辐照损伤, 发现制备工艺和强化相含量是影响钨基面向等离子体材料力学性能的主要方面, 而均匀分散的强化相颗粒所致使的组织致密化程度更高是钨基材料力学性能提高的主要因素。其次, 阐述了晶界和晶内的强化相颗粒分散不均表现出的位移损伤、气泡、绒毛、微裂纹等缺陷都将增加材料对氢同位素的捕获几率, 以及等离子体辐照造成的脆化硬化将降低材料的抗热冲击性能。最后分析了近些年弥散强化钨基面向等离子体材料存在的关键基础问题, 展望了未来弥散强化钨基材料的主要发展趋势, 期望为开发优异的抗高热负荷和辐照损伤的钨基材料方面提供重要参考。

**关键词:** 面向等离子体材料; 弥散强化; 钨基材料; 力学性能; 氢滞留; 辐照损伤

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## Plasma Facing Materials Based on Dispersion Strengthened Tungsten

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**ABSTRACT:** Plasma facing materials in extreme environment of fusion reactors suffer high temperature corrosion, irradiation damage, and severe fuel retention. Tungsten is currently the main candidate for plasma materials because of its inherent low thermal expansion coefficient, low tritium storage capacity, high radiation resistance, and good corrosion resistance. However, the interaction between plasma and tungsten under deuterium-tritium reaction exhibits such defects as low temperature brittleness, low recrystallization temperature and high toughness-brittle transition temperature, such as pores, vacancies and fatigue cracks, which are difficult to meet the wall loading requirements of future nuclear fusion reactors. At present, the second phase dispersion strengthening has become one of the common methods to improve the performance of tungsten-based plasma facing materials.

However, the existence of high heat load and high flux of particles put forward more stringent requirements for the comprehensive performance of dispersion strengthened tungsten-based materials. In recent years, the research on tungsten-based

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plasma-facing materials in fusion reactor has been mainly focused on three aspects: first, the effect of adding oxide ( $\text{Y}_2\text{O}_3$ ,  $\text{La}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ ) or carbide ( $\text{TiC}$ ,  $\text{TaC}$ ,  $\text{ZrC}$ ) second-phase particles on the microstructure and mechanical properties of tungsten-based materials; second, the hydrogen retention characteristics of dispersion strengthened tungsten based materials; third, the irradiation damage behavior under the action of different particle flows. The research on the mechanical properties of plasma tungsten based materials mainly includes grain size, tensile and compressive strength, recrystallization temperature, toughness brittleness transition temperature and microhardness. The material preparation process and strengthening phase particle content are the two main aspects affecting the mechanical properties of dispersion strengthened tungsten based materials. At present, the preparation methods of particle strengthening phase mainly include powder metallurgy, mechanical alloying, hydrothermal synthesis and wet chemical method, and the relative density and mechanical properties of tungsten based materials prepared by combining with different sintering methods are very different. The uneven dispersion of strengthening phase particles at grain boundaries and in grains, and the irradiation effect of the high-beam particles on the dispersion-strengthened tungsten-based material causes displacement damage, bubbles, fluff in the microstructure and morphology, micro-cracks and other defects will increase the probability of the material capturing hydrogen isotopes. In addition, the fusion reactor plasma irradiation will also cause embrittlement and hardening of the tungsten material, significantly reducing the thermal shock resistance of the material.

This paper reviews the effects of carbide or oxide content on the microstructure and mechanical properties of tungsten based plasma-facing materials prepared by different processes, as well as the latest research progress on hydrogen retention characteristics and irradiation damage in dispersion strengthened tungsten based materials at home and abroad, analyzes the key basic problems of dispersion-strengthened tungsten based plasma-facing materials in recent years, and prospects the main development trends of dispersion-strengthened tungsten based materials in the future, which is expected to provide an important reference for the development of tungsten based materials with excellent resistance to high thermal load and irradiation damage.

**KEY WORDS:** plasma facing materials; dispersion strengthening; tungsten-based materials; mechanical properties; hydrogen retention; irradiation damage

面向等离子体材料 (Plasma Facing Materials, PFMs) 是氘-氘聚变反应堆运行过程中高约束稳定运行的保证, 需承受芯部等离子体引起的高热流冲击和高通量粒子辐照<sup>[1]</sup>。为了满足未来聚变反应堆壁材料的服役要求, 面向等离子体材料 (PFMs) 必然要具有优异的力学性能和抗辐照损伤性能<sup>[2]</sup>。钨因其熔点高、低物理溅射率、低氚滞留率和良好的耐蚀性而被认为是聚变装置中面向等离子体材料 (PFMs) 的主要候选材料, 但纯钨存在再结晶温度低 (1 200 °C)、低温脆性和中子辐射脆化等问题难以达到理想的第一壁材料性能<sup>[3-5]</sup>。目前, 国内外主要采用以添加弥散强化相颗粒 (氧化物  $\text{Y}_2\text{O}_3$ 、 $\text{La}_2\text{O}_3$ 、 $\text{Al}_2\text{O}_3$ 、 $\text{ZrO}_2$  或碳化物  $\text{TiC}$ 、 $\text{TaC}$ 、 $\text{ZrC}$ )、掺杂元素合金化 ( $\text{Ta}$ 、 $\text{Re}$ 、 $\text{V}$ 、 $\text{Ni}$ )、纤维增韧强化等手段改善钨基材料的微观组织和综合性能<sup>[6-9]</sup>。相比之下, 弥散强化下的钨基材料具有更加优异的高温力学性能、高抗辐照损伤和低氢同位素滞留能力, 以及制备工艺简单等诸多优点而受到广泛关注<sup>[10-14]</sup>。

20 世纪 80 年代以来, 先进实验超导托卡马克 (EAST)、国际热核聚变实验堆 (ITER) 和中国聚变工程实验堆 (CFETR) 对未来聚变反应堆长脉冲高参数稳态运行的钨基面向等离子体材料开展了大量研究工作<sup>[15-16]</sup>。其中以添加氧化物 ( $\text{Y}_2\text{O}_3$ 、 $\text{La}_2\text{O}_3$ 、

$\text{Al}_2\text{O}_3$ 、 $\text{ZrO}_2$ ) 或碳化物 ( $\text{TiC}$ 、 $\text{TaC}$ 、 $\text{ZrC}$ ) 第二相颗粒对钨基材料的晶粒尺寸、抗拉抗压强度、再结晶温度、韧性-脆性转变温度和显微硬度的影响规律为主要研究内容, 等离子体与壁相互作用 (Plasma-Wall Interactions, PWI) 而引起的氢同位素滞留特性和辐照损伤行为研究同样对开发新型防护涂层材料具有重大意义。

## 1 弥散强化钨基材料的力学性能

普遍认为, 第二相以极细小的颗粒形态均匀分布于钨基材料中, 增加相界面积, 阻碍晶界位错的运动, 显著提高钨合金的显微硬度、延展性、抗拉抗压强度<sup>[17-19]</sup>。同时, 弥散强化形成的高致密化程度钨基材料还表现出低韧性-脆性转变温度、高热稳定性、高抗热震能力和再结晶温度, 已逐渐成为聚变反应堆中主要的第一壁材料<sup>[20-21]</sup>。目前, 弥散强化钨基材料主要是以氧化物 ( $\text{Y}_2\text{O}_3$ 、 $\text{La}_2\text{O}_3$ 、 $\text{Al}_2\text{O}_3$ 、 $\text{ZrO}_2$ ) 或碳化物 ( $\text{TiC}$ 、 $\text{TaC}$ 、 $\text{ZrC}$ ) 作为超细晶第二相分散剂, 但不同制备工艺、固结方法和强化相含量将对钨基材料的力学性能造成显著差异<sup>[22]</sup>。如表 1 列出了常见粉末制备和固结方法下氧化物弥散强化钨合金的组织

表 1 氧化物弥散强化钨合金的微观组织和力学性能  
Tab.1 Microstructure and mechanical properties of oxide dispersion strengthened tungsten alloy

Methods	Alloy	Process	Grain size/ $\mu\text{m}$		[Density/( $\text{g}\cdot\text{cm}^{-3}$ )]/ (RD/%)	Microhardness (HV)	Refs.
			W grain	Oxide particle			
Mechanically alloyed	W-1%La <sub>2</sub> O <sub>3</sub>	HIP	—	>5	18.9/90.6	—	[23]
	W-1%Y <sub>2</sub> O <sub>3</sub>	Sintering	4	1.4	—/<18.5	412.7	[24]
	W-1%Y <sub>2</sub> O <sub>3</sub> (0.2%Zr)	Swaging	4.7	0.1~2	—/99.3	467	[25]
	W-5%HfO <sub>2</sub>	SPS	11.6	>5	—/94.5	440	[26]
Hydrothermal method	W-1.5%ZrO <sub>2</sub>	SPS	4.65	2.5	—/98.9	538.8	[27]
	W-6vol% Al <sub>2</sub> O <sub>3</sub>	SPS	3.64	>1.0	—/94.96	347.39	[28]
	W-0.75%Zr(Y)O <sub>2</sub>	HIP	4.3	0.38	18.31/96.5	486	[29]
	W-1wt%Y <sub>2</sub> O <sub>3</sub>	LST	0.21	<0.05	—/98.0	726	[30]
	W-0.5wt%Y <sub>2</sub> O <sub>3</sub>	LST	0.2	<0.15	—/97.8	732.6	[31]

## 1.1 氧化物弥散强化钨基材料

### 1.1.1 W-Y<sub>2</sub>O<sub>3</sub>

稀土氧化物可有效地提高钨基材料的低温脆性和再结晶脆性,原因在于均匀分散在钨晶粒内部的超细氧化物颗粒可以产生、固定和积累位错,进一步增强材料的力学性能,而晶界处的氧化物颗粒会阻碍晶界迁移,有效抑制晶粒的生长<sup>[32]</sup>。Y<sub>2</sub>O<sub>3</sub> 具有高熔点(2 690 ℃)、理化性质稳定等优点,被认为高性能、超细晶弥散强化钨基材料的首选强化相<sup>[33]</sup>。

针对相同氧化物颗粒强化,不同制备工艺及配比对其力学性能有着极大影响。普遍发现,粉末冶金、湿化学以及溶胶-凝胶法等能显著提高氧化物弥散强化钨基材料的显微硬度和延展性,降低孔隙率。粉末冶金与热锻的 W-2wt.%Y<sub>2</sub>O<sub>3</sub> 合金中的晶粒含有少量位错现象,Berkovich 硬度和杨氏模量分别为 4.9、400 GPa,DBTT 值在 773~873 K 内。与机械合金化条件下的 W-2%Y<sub>2</sub>O<sub>3</sub> 相比,673~1 273 K 的 DBTT 值表现出更加优异的延展性<sup>[34]</sup>。Lian 等<sup>[35]</sup>研究了常规烧结和高热能锻造的 W-Y<sub>2</sub>O<sub>3</sub> 的热力学特性。与常规烧结相比,高能锻造下的 W-Y<sub>2</sub>O<sub>3</sub> 材料的抗拉强度与延展性有所提升,200 ℃下的 HERF W-Y<sub>2</sub>O<sub>3</sub> 的抗拉强度为 948 MPa,而轧制的 W-Y<sub>2</sub>O<sub>3</sub> 为 846 MPa,并且热冲击开裂阈值低于 100 ℃。采用新型冷却干燥和低温烧结法制备钨晶粒尺寸为 410 nm 的 W-Y<sub>2</sub>O<sub>3</sub> 合金,

发现 Y<sub>2</sub>O<sub>3</sub> 颗粒能够吸收自由氧杂质,与钨基体反应形成第二相 Y<sub>2</sub>WO<sub>6</sub> (WO<sub>3</sub>·Y<sub>2</sub>O<sub>3</sub><20 nm) 颗粒或钨基体界面的 W-Y-O 三元相扩散层。Y<sub>2</sub>WO<sub>6</sub> 粒子与钨形成具有共格或半共格结构材料,显微硬度高达 (690.2±32.0)HV<sub>0.2</sub><sup>[36]</sup>。Dong 等<sup>[37]</sup>报道了氢气气氛下低温烧结制备的 W-Y<sub>2</sub>O<sub>3</sub> 合金,发现氧化物颗粒的增加使得体积分数和晶间与晶内氧化物颗粒的尺寸减小,在 700 ℃时,W-Y<sub>2</sub>O<sub>3</sub> 合金的强度高达 1 134 MPa,总伸长率超过 30%。然而,相同成分的 W-Y<sub>2</sub>O<sub>3</sub> 复合材料在不同温度下的力学性能同样具有差异性,见表 2。其原因可能是温度在 300 ℃上时,材料的晶粒不断长大,发生再结晶脆性。

此外,添加 V、Ta、Ni、Ti 和 Zr 元素可以改善 W-Y<sub>2</sub>O<sub>3</sub> 的致密化程度、表面形貌和力学性能,提高抵抗辐照脆化硬化能力。采用机械合金化分别制备了含有 V 和 Ta 元素的原位分散氧化钨钨基材料。发现含 V 元素材料具有分散均匀的超细第二相颗粒,Ta 能使不规则形状的 Y<sub>2</sub>O<sub>3</sub> 颗粒更具球形,并且显微组织主要呈现 TaVO<sub>4</sub> 和 V<sub>2</sub>O<sub>5</sub> 相,而 Y<sub>2</sub>O<sub>3</sub> 与 Ta<sub>2</sub>O<sub>5</sub> 相互作用形成 YT<sub>2</sub>O<sub>4</sub> 相<sup>[40]</sup>。类似的,Daoush 等<sup>[6]</sup>对含有 5% (质量分数) Ni 和 Y<sub>2</sub>O<sub>3</sub>、ZrO<sub>2</sub> 或 TiO<sub>2</sub> 弥散强化相的微观结构和力学性能进行了研究。结果表明,与 W-Ni-ZrO<sub>2</sub> 和 W-Ni-TiO<sub>2</sub> 相比,W-Ni-Y<sub>2</sub>O<sub>3</sub> 合金具有相对均匀的互联结构,更高的致密化程度、抗压强度和更低的孔隙率,而 W-Ni-TiO<sub>2</sub> 具有最高的维氏硬度

表 2 不同 W-1.0wt%Y<sub>2</sub>O<sub>3</sub> 复合材料的拉伸性能与温度的关系<sup>[37]</sup>  
Tab.2 Relationship between tensile properties and temperature of different W-1.0wt% Y<sub>2</sub>O<sub>3</sub> composites<sup>[37]</sup>

Materials	(Tensile strength/MPa)/(Elongation/%)						Refs.
	RT	200 ℃	300 ℃	400 ℃	500 ℃	600 ℃	
SPSed W-1.0wt%Y <sub>2</sub> O <sub>3</sub>	263/—	—	—	484/—	538/3.6	430/8.8	[38]
Swaged W-1.0wt%Y <sub>2</sub> O <sub>3</sub>	517/—	668/0.7	577/12	501/18	489/13.6	412/12	[38]
A-swaged W-1.0wt%Y <sub>2</sub> O <sub>3</sub>	482/—	842/6.4	579/20.8	480/16	—	—	[38]
SPSed sol-gel W-1%Y <sub>2</sub> O <sub>3</sub>	—	—	632/1.6	572/11.2	522/16.8	478/15.2	[39]
Swaged sol-gel W-1%Y <sub>2</sub> O <sub>3</sub>	—	619/1.6	506/13.6	497/14.8	503/15.6	478/14.2	[39]

值。活性元素 Zr 对 C、O、N 等杂质元素具有很高的亲和力,能够降低 C 和 O 等气体间隙元素在晶界产生的缺陷。例如,当试验温度从 300 ℃ 提高到 600 ℃ 时,W-0.2%Zr-1%Y<sub>2</sub>O<sub>3</sub> 的极限拉伸强度从 593 MPa 逐渐下降到 478 MPa,总伸长量从 9.8% 上升到 16.4%。在 500 ℃ 以上时,W-0.2%Zr-1%Y<sub>2</sub>O<sub>3</sub> 合金的强度和延展性均高于 W-1%Y<sub>2</sub>O<sub>3</sub>, 并发现 ZrH<sub>2</sub> 脱氢后, Zr 与晶界处氧先发生反应形成 ZrO<sub>2</sub>, 再与 Y<sub>2</sub>O<sub>3</sub> 颗粒结合成 Y-Zr-O 氧化物粒子,表明微量 Zr 的晶界强化可以有效提高 W-1%Y<sub>2</sub>O<sub>3</sub> 的低温延性和抗拉强度<sup>[27]</sup>。此外,有研究表明,真空烧结制备的 W-0.2%Zr-1%Y<sub>2</sub>O<sub>3</sub> 由 100 ℃ 上升至 200 ℃ 时,材料的塑性和极限拉伸强度都在不断提高,该合金的韧性-脆性转变温度表现为 150 ℃<sup>[41]</sup>。因此,晶粒细化是提高合金力学性能的关键因素。

1.1.2 W-La<sub>2</sub>O<sub>3</sub>

由于处于聚变反应堆的面向等离子体材料(PFMs)需承受等离子体破坏带来的瞬态高热流,而钨材料在一定程度上存在再结晶温度低和韧脆转变温度高的性能,无法进一步满足未来聚变反应堆的壁载要求<sup>[42]</sup>。研究表明,La<sub>2</sub>O<sub>3</sub> 的加入可以明显细化钨晶粒,改善其力学性能<sup>[43]</sup>。例如,采用微/纳米复合工艺制备的 W-La<sub>2</sub>O<sub>3</sub> 材料能够承受 200 MW/m<sup>2</sup> (5 ms) 的高温热流,并且当 La<sub>2</sub>O<sub>3</sub> 的质量分数为 0.3% 时,W 材料的弯曲强度达到最高。不同 La<sub>2</sub>O<sub>3</sub> 含量的 W-La<sub>2</sub>O<sub>3</sub> 复合材料的组织和力学性能见表 3<sup>[44]</sup>。Qin 等<sup>[45]</sup>采用湿化学方法制备了相对密度为 95.0% 的 W-La<sub>2</sub>O<sub>3</sub> 复合材料,经晶界和晶内的 La<sub>2</sub>O<sub>3</sub> 氧化物颗粒强化,W-La<sub>2</sub>O<sub>3</sub> 复合材料的显微硬度和抗压强度分别高达 684.0HV<sub>0.2</sub> 和 2 119 MPa,进一步证明晶粒细化是提高材料力学性能的关键。

表 3 不同 La<sub>2</sub>O<sub>3</sub> 含量的 W-La<sub>2</sub>O<sub>3</sub> 材料性能<sup>[44]</sup>  
Tab.3 Properties of W-La<sub>2</sub>O<sub>3</sub> materials with different La<sub>2</sub>O<sub>3</sub> contents<sup>[44]</sup>

	Theoretical density/(g·cm <sup>-3</sup> )	Relative density/%	Grain size/μm	Bending strength/MPa
W-0.1wt%La <sub>2</sub> O <sub>3</sub>	19.31	99.1	10~15	325
W-0.3wt%La <sub>2</sub> O <sub>3</sub>	19.24	99.5	8~12	550
W-0.5wt%La <sub>2</sub> O <sub>3</sub>	19.16	98.5	7~10	460
W-0.7wt%La <sub>2</sub> O <sub>3</sub>	19.09	98.9	5~8	500

1.1.3 W-Al<sub>2</sub>O<sub>3</sub>

与 Y<sub>2</sub>O<sub>3</sub> 和 La<sub>2</sub>O<sub>3</sub> 氧化物相比,Al<sub>2</sub>O<sub>3</sub> 以其高硬度、低密度、优异的热化学稳定性和良好的耐磨耐腐蚀性得到广泛应用<sup>[46-47]</sup>。Wang 等<sup>[30]</sup>采用水热合成法和放电等离子体烧结相结合,将 Al<sub>2</sub>O<sub>3</sub> 超细颗粒添加到钨基体中,研究了放电等离子体烧结温度对 W-Al<sub>2</sub>O<sub>3</sub>

复合材料致密化的影响。研究表明,1 800~2 100 ℃ 烧结温度的 W-Al<sub>2</sub>O<sub>3</sub> 复合材料的相对密度<92%,在 1 900~2 000 ℃ 时达到最大值 97%,并且随温度的升高,晶粒尺寸从 2.49 μm 增加到 8.21 μm。此外,相同制备方法下,不同的 Al<sub>2</sub>O<sub>3</sub> 成分含量对 W-Al<sub>2</sub>O<sub>3</sub> 复合材料组织与力学性能的影响见表 4。由于 W-6% Al<sub>2</sub>O<sub>3</sub> (Al<sub>2</sub>O<sub>3</sub> 的体积分数为 6%) 中的氧化物颗粒均匀分布,具有最优异的力学性能,平均钨晶粒尺寸和显微硬度值分别为 2.78 μm 和 435.56HV<sub>200 g</sub>。同样,采用水热合成法制备了 Al<sub>2</sub>O<sub>3</sub> 质量分数为 0~1% 的 W-Al<sub>2</sub>O<sub>3</sub> 复合材料,结果发现,Al<sub>2</sub>O<sub>3</sub> 质量分数达到 0.25% 时,显微硬度和抗压强度分别达到最大值 386HV 和 1628 MPa。根据 W-25%Al<sub>2</sub>O<sub>3</sub> 的热变形活化能  $Q=321.79$  kJ/mol,进一步给出合金的热变形本构方程,如式(1)所示<sup>[48]</sup>。

$$\dot{\epsilon} = 20.7829 [\sinh(0.0020 \sigma)]^{27.2500} \exp(-321790/RT) \quad (1)$$

式中:  $\dot{\epsilon}$  为合金的应变速率, s<sup>-1</sup>;  $\sigma$  为峰值应力或稳态应力, MPa;  $R$  为气体常数, J/(mol·K);  $T$  为热力学温度, K。可以利用应力-应变曲线峰值和稳态应力来评估材料的流动应力引起的高温变形量。

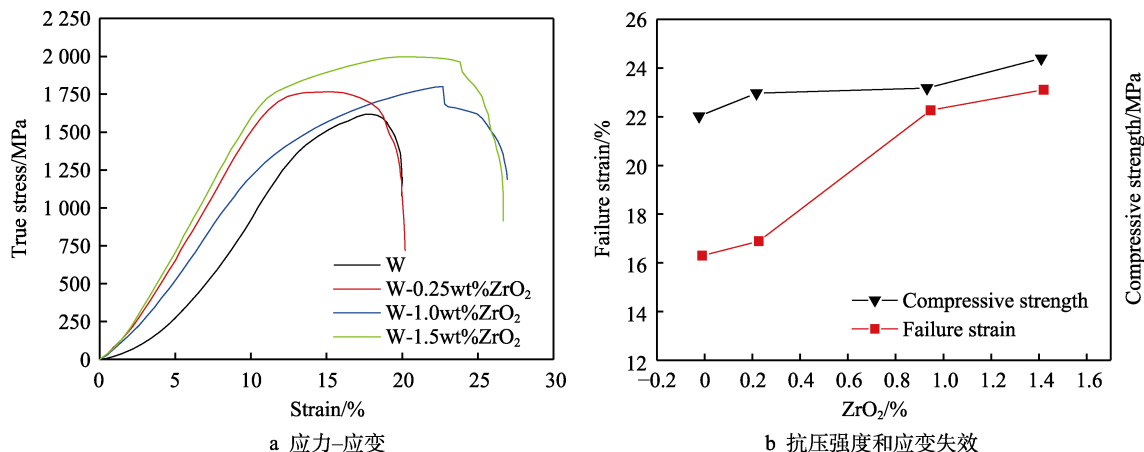
表 4 不同 Al<sub>2</sub>O<sub>3</sub> 成分含量的 W-Al<sub>2</sub>O<sub>3</sub> 复合材料组织与力学性能<sup>[30]</sup>

Tab.4 Microstructure and mechanical properties of W-Al<sub>2</sub>O<sub>3</sub> composites with different Al<sub>2</sub>O<sub>3</sub> content<sup>[30]</sup>

Composition	Relative density/%	Microhardness (HV <sub>200 g</sub> )	Grain size/μm
Pure W	90.65	277.79	4.25
W-2vol%Al <sub>2</sub> O <sub>3</sub>	94.96	347.39	3.64
W-4vol%Al <sub>2</sub> O <sub>3</sub>	95.69	366.06	3.08
W-6vol%Al <sub>2</sub> O <sub>3</sub>	96.83	435.56	2.78
W-8vol%Al <sub>2</sub> O <sub>3</sub>	96.56	396.10	6.15
W-15vol%Al <sub>2</sub> O <sub>3</sub>	95.15	342.75	8.46

1.1.4 W-ZrO<sub>2</sub>

ZrO<sub>2</sub> 通常为脆性金属间化合物的增韧强化相颗粒,是因为本身是具有高硬度、高熔点、耐腐蚀性以及优异的热特性材料。研究表明,高温的 ZrO<sub>2</sub> 冷却到室温时,将从立方相转变为单斜相。Li 等<sup>[49]</sup>采用粉末冶金法制备的 W-xZrO<sub>2</sub> (x=0%、0.25%、1.0%、1.5%,质量分数)的微观结构表明,W-1.5%ZrO<sub>2</sub> 合金的平均晶粒尺寸为 47.18 μm,抗压强度和破坏应变分别高达 1941.3 MPa 和 23.1%,比纯钨分别提高 23.27% 和 22.37%,如图 1 所示。此外,对于采用水热法和等离子体烧结制备的 W-ZrO<sub>2</sub> 复合材料,研究发现,ZrO<sub>2</sub> 成分含量对 W-ZrO<sub>2</sub> 复合材料的综合性能影响与文献[49]研究的结果类似,W-1.5%ZrO<sub>2</sub> 复合材料的综合性能最优,显微硬度为 538.8HV,当室温压缩至应变失效时的抗压强度为 1 628 MPa,应变率为 1.7×10<sup>-3</sup> s<sup>-1</sup><sup>[29]</sup>。

图1 不同 ZrO<sub>2</sub> 含量的合金力学性能<sup>[49]</sup>Fig.1 Mechanical properties of alloys with different ZrO<sub>2</sub> contents<sup>[49]</sup>:

a) stress-strain; b) compressive strength and strain failure

## 1.2 碳化物弥散强化钨基材料

### 1.2.1 W-TiC

为了缓解高温下第一壁材料的腐蚀和辐射损伤, TiC 弥散强化钨基材料以其高抗弯强度、良好的冲击延展性、优异的高温抗拉强度和抗辐照硬化性能而被广泛关注<sup>[50-51]</sup>。W-TiC 暴露在 1.65~1.75 MJ/m<sup>2</sup> 热流中 5 ms 内才开始发生裂纹,而在 1 000 脉冲下 0.55 GW/m<sup>2</sup> 瞬态热负荷中表现出明显的脆性断裂特征<sup>[52]</sup>。Miao 等<sup>[53]</sup>利用粉末冶金和热轧工艺相结合的方法制备了 W-0.5%TiC 和 W-0.5%TiC 合金,这 2 种合金在 1 400~1 600 °C 具有等温退火热稳定性。研究表明,热轧制的 W-0.5%TiC 合金在 200 °C 下的强度为 789 MPa,总伸长率为 4.8%,合金在 1 600 °C 退火 1 h 后的力学性能明显下降,比轧制纯钨高 250 °C,并且发现 TiC 弥散强化结构在 1 200 °C 的使用环境下具有优异的热稳定性。此外,Lang 等<sup>[54]</sup>研究了湿化学和放电等离子体烧结的制备工艺下不同 TiC 含量(质量分数 0~0.9%)对 W-TiC 合金组织、力学性能和热传导的影响,见表 5。研究表明,W-0.5%TiC 合金的力学性能相对最优,在常温和 800 °C 下的热导率分别为 139.6、105.4 W/(m·K);TiC 的质量分数为 0.1%~0.5% 的 W-TiC 合金,其热导率略低于纯钨;TiC 的质量分

数为 0.7%和 0.9%的 W-TiC 合金,力学性能和导热性能有明显下降,可能是晶界上的 TiC 颗粒不均匀聚集所导致的。

### 1.2.2 W-TaC

与 La<sub>2</sub>O<sub>3</sub> 和 Y<sub>2</sub>O<sub>3</sub> 相比, TaC 具有更高的熔点(3 880 °C)和热稳定性,同时 TaC 弥散强化能有效提高钨合金的抗冲蚀性和再结晶温度<sup>[55]</sup>。研究表明,W-0.5%Ta-0.01%C 合金中的 TaC 颗粒提高了钨材料的延展性,能与氧杂质在晶界形成 Ta-C<sub>x</sub>-O<sub>y</sub> 化合物,降低自由氧浓度而提高晶界强度,并在 1 600 K 下具有良好的热稳定性<sup>[56]</sup>。Miao 等<sup>[57]</sup>报道了一种在 200 °C 时极限抗拉强度为 982 MPa 的细晶 W-0.5%TaC 合金材料,在 RT~500 °C 的极限抗拉强度均高于 570 MPa,并发现存在大量位错现象。此外,Xie 等<sup>[58]</sup>研究了 TaC 对钨材料拉伸性能和热稳定性的影响。结果表明,TaC 阻碍晶界迁移、扎钉位错和细化晶粒,W-1.0% TaC 的再结晶温度约为 1 400 °C,轧制的 W-1.0%TaC 在室温下的裂纹阈值为 0.33~0.44 GW/m<sup>2</sup>,而存在的 Ta<sub>2</sub>O<sub>5</sub> 和 Ta-C<sub>x</sub>-O<sub>y</sub> 颗粒将导致 W-TaC 的抗热冲击性能下降。

### 1.2.3 W-ZrC

与 La<sub>2</sub>O<sub>3</sub> 强化相相比, ZrC 具有更高的熔点

表 5 不同 TiC 含量的 W-TiC 组织和力学性能<sup>[54]</sup>Tab.5 Microstructure and mechanical properties of W-TiC with different TiC content<sup>[54]</sup>

Alloy	Average grain size/ $\mu\text{m}$	Average TiC particle size/nm	Relative density/%	Micro hardness (Hv <sub>0.2</sub> )	Bending strength/MPa	Flexural strain/%
PW	2.16	—	95.44	469	645.88	0.84
W-0.1wt%TiC	1.76	50	96.58	633.3	812.84	1.16
W-0.3wt%TiC	1.34	60	97.31	670.7	921.12	1.19
W-0.5wt%TiC	0.91	62	97.61	739.1	1 065.72	1.23
W-0.7wt%TiC	0.71	71	94.99	843.3	822.47	0.84
W-0.9wt%TiC	0.53	97	93.72	843.68	843.68	0.9



(3 540 °C), 并作为吸气剂减少氧对壁材料的影响。研究表明, ZrC 弥散强化的钨基复合材料表现出高强度、高延展性、低韧脆转变温度、热稳定性以及抗热震性等优异的力学性能<sup>[59-61]</sup>。例如, W-0.5%ZrC 合金在室温下的开裂阈值为 220~330 MW/m<sup>2</sup>, 相当于 7.0~10.4 MJ/(m<sup>2</sup>·s<sup>1/2</sup>) 热负荷<sup>[62]</sup>。Xie 等<sup>[63]</sup>采用粉末冶金和放电等离子体烧结制备了 W-(0.2,0.5,1.0)%ZrC 复合材料。发现材料的韧脆转变温度均在 500~600 °C 内, W-0.5% ZrC 合金在 700 °C 时的极限抗拉强度为 535 MPa, 总伸长率为 24.8%, 而 ZrC 质量分数大于 0.5% 时的伸长率将会有所下降。相同温度下, W-ZrC 合金的拉伸性能随 ZrC 含量的增加而上升, 但显微硬度没有明显变化趋势。ZrC 可以与杂质氧形成立方 Zr-C-O 和单斜 ZrO<sub>2</sub> 相, 减少氧对晶界强度的影响。此外, 一种再结晶温度为 1 800 °C 以及韧脆转变温度在 400~500 °C 内的 W-0.5%ZrC-1%Re 复合材料被报道出来, 发现该材料在 700 °C 下的极限拉伸强度和总伸长率分别是纯钨的 1.3、2.6 倍<sup>[64]</sup>。热轧制的 W-0.5%ZrC 在 1 500 °C 下的极限拉伸强度值最高, 并表现出晶界强化和第二相颗粒 2 种强化现象<sup>[65]</sup>。

## 2 弥散强化钨基材料的氢滞留特性

针对钨基面向等离子体材料的力学性能问题, 国内外研究学者主要是采用从掺杂组元的合金化、弥散强化以及制备纳米钨等方面来改善。然而, 除了优良的力学性能, 减少燃料滞留也是目前钨基面向等离子体材料的首要研究课题, 特别是氧化物/碳化物弥散强化下钨基材料中的氢滞留行为<sup>[66]</sup>。

### 2.1 氧化物弥散强化钨基材料中的氢滞留

氧化物强化相颗粒优化提高了钨基材料的组织和力学性能, 但晶界与晶内的氧化物颗粒分散不均, 将增加更多氢类的捕获点, 是因为氢滞留与材料的晶粒尺寸、织构、杂质和缺陷密度等具有很强的相关性。例如, Ti 掺杂的超细晶粒钨中的 TiO<sub>2</sub> 粒子存在, 发现了更多的 D 俘获位点<sup>[67]</sup>。Tan 等<sup>[68]</sup>研究了高能锻造的 W-0.5%Y<sub>2</sub>O<sub>3</sub> 合金暴露在温度为 443~463 K、离子通量 1.2×10<sup>22</sup> D/(m<sup>2</sup>·s) 下的氘滞留情况。结果表明, 横截面在 0.5~3 h 辐照时间的氘滞留从 1.28×10<sup>19</sup> D/m<sup>2</sup> 增加到 4.96×10<sup>21</sup> D/m<sup>2</sup>, 而锻造面的氘总滞留 (2.25×10<sup>20</sup>~9.73×10<sup>20</sup> D/m<sup>2</sup>) 却无明显变化, 但产生严重的气泡现象。另外, 利用核反应分析和热解析光谱研究了掺杂 Y<sub>2</sub>O<sub>3</sub> 颗粒的钨合金在入射通量为 6×10<sup>24</sup>、7.5×10<sup>25</sup> D/m<sup>2</sup> 和样品温度为 370、450、570 K 下的氘滞留行为<sup>[69]</sup>。研究表明, 500、370、450 K 的 W-Y<sub>2</sub>O<sub>3</sub> 局部氘浓度与纯钨相差 20%~30%, W-Y<sub>2</sub>O<sub>3</sub> 合金中的总氘浓度比纯钨高约为 3.6 倍。这是因为掺杂 Y<sub>2</sub>O<sub>3</sub> 颗粒的钨合金存在本征缺陷, 具有很高的捕获能, 亚微米孔隙使得高温氘释放, 并且氘滞留量随

样品温度的升高有所增加, 在一定温度下达到峰值。

### 2.2 碳化物弥散强化钨基材料中的氢滞留

由于碳空位可以有效地捕获氢同位素, 氢在碳化物弥散强化钨基材料中的滞留差异与碳化物析出有关。例如, W-1.1TiC 和 W-3.3TaC 中的碳化物析出可能提供了大量的氢捕获点<sup>[70]</sup>。Oya 等<sup>[71]</sup>研究了增韧细晶再结晶钨合金 (Toughened, Fine-Grained Recrystallized, TFGR W-1.1%TiC) 中的氘滞留, 其中钨样品中氢保率与辐照温度的关系如图 2 所示, TFGR W-1.1%TiC 合金在 473 K 时的氘滞留率水平与纯钨基本相同。当 T>473 K 时, TFGR W-1.1%TiC 合金的氘滞留量均高于纯钨, 与 D 辐照相比, 473 K 下 D+He 辐照的 TFGR W-1.1%TiC 合金的氘滞留降低了 70%。同时, NRA 分析出纯钨中的大部分氘原子是停留在近表层, 深度>1 μm 的氘原子分数低于 0.001%, 而 TFGR W-1.1%TiC 合金厚度>1 μm 的氘滞留比纯钨高 0.004%。表明 TiC 致使钨合金中氘滞留量增加的可能原因是组织结构中存在晶界扩散 (TiC 分散体作为附加捕集点) 和 TiD<sub>2</sub> 形成。类似的, 利用核反应分析和热解析光谱对暴露在 573 K 温度, 注入量 1×10<sup>26</sup> D/m<sup>2</sup> 下的增韧细晶再结晶钨合金 (TFGR W-1.2%TiC 和 TFGR W-3.3%TaC) 中的氘滞留情况进行测量。发现 W-TiC 在 0~8 μm 深度内 D 的平均滞留量为 1.3×10<sup>21</sup> D/m<sup>2</sup>, W-TaC 中为 1.4×10<sup>21</sup> D/m<sup>2</sup>。同时, TDS 测得 W-TiC 的氘总滞留量为 3.9×10<sup>21</sup> D/m<sup>2</sup>, W-TaC 为 3.0×10<sup>21</sup> D/m<sup>2</sup>, 并且 W-TiC 和 W-TaC 均出现氘的解吸峰。W-TaC 比 W-TiC 中氘滞留率低的原因是 Ta 和 Ti 对氘原子的化学亲和力不同<sup>[72]</sup>。

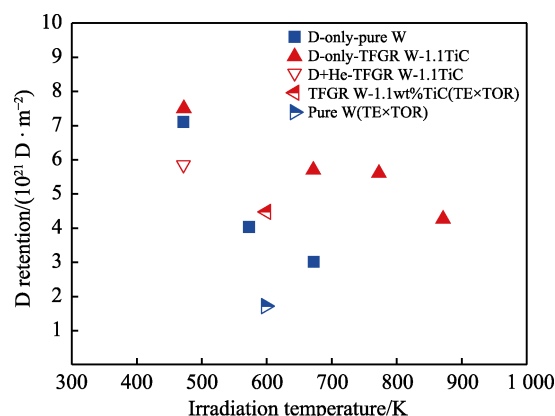


图 2 钨样品中 D 总滞留与辐照温度的热解析光谱图<sup>[72]</sup>  
Fig.2 Thermal analysis spectrum of total D retention and irradiation temperature in tungsten sample<sup>[72]</sup>

Zibrov 等<sup>[73]</sup>利用核反应分析和热解析光谱测量了 W-1.1%TiC 和 W-3.3%TaC 合金样品在 800~963 K、100 kPa 压力下暴露于 D<sub>2</sub> 气体中的氘含量。发现 W-3.3%TaC 合金中的氘体积浓度和总滞留量均与纯多晶钨大致相同, 而 W-1.1%TiC 合金中的氘体积浓



粒尺寸增加,但强化相颗粒的存在可以大大降低钨材料的辐照损伤。

## 4 结语

钨基面向等离子体材料与高热负荷和高能粒子流相互作用下的综合材料性能要求,是限制先进聚变反应堆高约束参数稳定运行的关键技术难题之一。目前,国内外学者采用弥散强化手段来改善钨基面向等离子体材料的微观组织与力学性能、抗辐照损伤以及氢滞留行为,但当前已开发的弥散强化钨基材料与未来聚变反应堆极端工况下的材料性能仍存在一定差距,主要问题包括以下几个方面:对于添加第二相强化相颗粒无法均匀分散在钨基材料晶界,易导致部分区域开裂;弥散强化钨基材料中存在大量孔隙,界面结合力较低;晶界处的部分晶体表现出高度无序状态和存在的 N、O 杂质易降低晶界内聚力,使材料延展性显著降低;粉末固结的弥散强化钨基材料中的残余内应力没有将其完全释放,易于在等离子体冲击下产生微裂纹。因此,弥散强化钨基材料的一些关键的基础问题和技术还需进一步深入探讨与研发。

1) 粉末制备工艺和烧结方法的创新与优化。探讨不同粉末烧结方法下的材料密度、孔隙、晶粒尺寸以及力学性能的变化规律,针对相同强化相颗粒及配比的单一烧结方法下产生材料缺陷,建立粉末粒度、烧结方法与温度、后处理工艺对材料综合性能的影响准则,得出最为优化的生产工艺。

2) 弥散强化钨基材料中的强化相种类及含量对力学性能和抗辐照损伤行为的影响规律及微观机制。明确强化相成分与组织性能的关系,建立聚变反应堆中用弥散强化钨基材料的成分设计准则,确定强化相类型及配比与材料力学性能和抗辐照损伤的调控机制,进而制备性能最为优异的弥散强化钨基材料。

3) 辐照对弥散强化钨基材料的微观结构缺陷的影响规律,如辐照粒子种类、辐照能量、粒子剂量、辐照温度等条件对弥散强化钨基材料的形貌特征与缺陷类型和数量之间的关系,深入了解不同粒子及协调作用下与增强相钨材料的相互作用机理,进一步设计出适用于聚变反应堆环境下的钨基材料。

4) 扩展新型强化相类型或添加元素使基体钨和强化相颗粒细化和分散均匀。探索微量元素 V、Ta、Ni、Ti 和 Zr 等对晶粒和氧化物/碳化物强化相颗粒尺寸的影响规律,确定元素种类及含量与钨基材料力学性能之间的关系,发展如氮化物(TiN)、硼化物、硅化物等作为新增强化相对钨基材料综合性能的影响。

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