

先进核能系统用 ODS 钢成分设计及制备工艺研究进展

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摘要 核能是一种清洁能源, 发展具有更高安全性和经济性的第四代核能系统是当下各国研究的热点。传统的锆合金已经无法适应第四代核能系统苛刻的服役环境, 开发具有优异综合性能的材料成为核能工程应用领域亟待解决的问题。氧化物弥散强化(Oxide dispersion strengthened, ODS)钢因具有良好的综合性能被认为是第四代核能系统反应堆包壳的重要候选材料, 是核材料领域研究热点。

ODS 钢优异的性能源于其合理的成分设计及独特的显微组织, 高数密度的弥散氧化物粒子极大的改善了合金的高温力学性能及抗辐照性能。尽管在世界各国围绕 ODS 钢在成分设计-性能-制备工艺开展了大量的研究工作, 但是在批量生产方面仍然存在挑战制约了 ODS 钢的工程应用。

本文就国内外关于 ODS 钢的显微组织、成分设计及制备技术等研究工作进行总结和分析, 对 ODS 钢的在核能领域的应用前景和当下存在的问题进行总结和展望, 为核级 ODS 钢的发展提供参考。

关键词 先进核能系统; ODS 钢; 显微组织; 成分设计; 制备工艺

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Research Progress On Composition Design And Preparation

Technology of ODS Steels For Advanced Nuclear Energy System

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ABSTRACT Developing the Gen IV nuclear energy system with higher safety and economy is the focus of current research as the nuclear energy is a clean energy. To develop the materials with excellent comprehensive properties has become the urgent problem as the traditional zirconium alloy was unable to adapt the harsh service environment of the Gen IV nuclear energy system. Oxide dispersion strengthened (ODS) steel is considered to be an important candidate clad material for the Gen IV energy system owing to its excellent comprehensive properties.

Reasonable composition design and unique microstructure are important reasons for the excellent properties of ODS steel. The high number density of dispersed oxide particles greatly improves the high temperature mechanical properties and irradiation resistance of the alloy. Although a lot of research work has been carried out around the world on the composition design-property-preparation process of ODS steels, there still exists challenges in mass production that limit the engineering applications of ODS steels.

This paper summarizes and analyzes the research work on microstructure, composition design and preparation technology of ODS steels in domestic and international, and summarizes and outlooks the application prospects and current problems of ODS steels in the field of nuclear energy, so as to provide reference for the

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development of nuclear-grade ODS steels.

KEY WORDS advanced nuclear energy systems; ODS steel; microstructure; composition design; preparation process

1.核电发展现状

核能因为低碳、高能量密度等特点而日益成为解决能源危机和环境问题的重要途径并受到广泛关注^[1, 2]。如图 1 所示^[3]，自上个世纪 50 年代人类和平利用核能以来，至今已经有 70 余年的历史。核裂变能与核聚变能是核能主要利用的两种形式。核聚变产生的能量远远大于裂变所释放的能量，而且氦、氦之间的产物无污染、无反射性。加之氦、氦在地球上储量丰富，因而核聚变被认为是一种清洁、高效并且无穷尽的理想能源。但核聚变能在商用的过程中仍然有许多技术问题尚未解决，因而技术研究较为成熟的核裂变堆率先得到商用。目前商业运营和正在建造的多为第二、三代裂变反应堆。

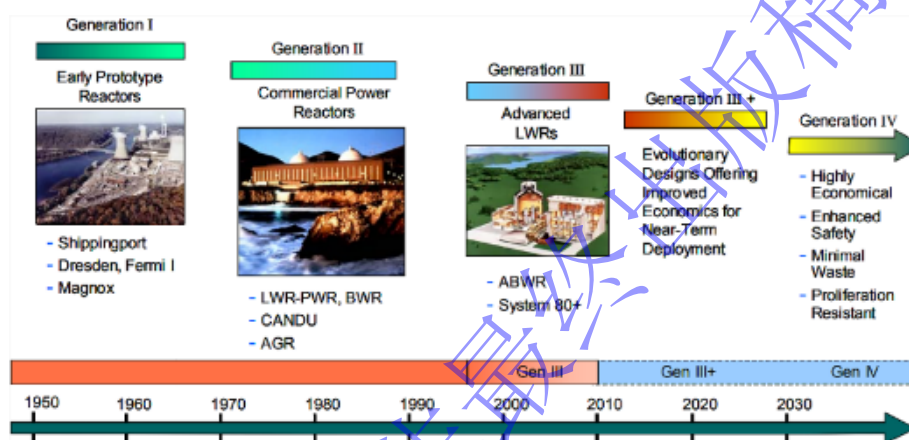


图 1 核能技术发展路线图^[3]

Fig.1 Nuclear Technology Roadmap^[3]

为提高当下核裂变堆的安全性与经济性，目前国内外正大力研究和发展第四代裂变堆。第四代核能系统的概念设计有如下六种：超临界水堆(SCWR)，钠冷快堆(SFR)，铅冷快堆(LFR)，气冷快堆(GFR)，超高温气冷反应堆(VHTR)，熔盐反应堆(MSR)。表 1^[4]列举了第四代反应堆工况条件及候选材料，通过服役工况对比可知，第四代堆的服役温度和中子辐照强度远远高于第二代轻水反应堆(LWR)^[5]。为能够适应服役环境的需要，核能系统燃料包壳材料必须至少同时具备以下性能^[6, 7]：(1)良好的高温强度及高温抗蠕变性能；(2)良好的耐蚀性；(3)良好的抗辐照性能；(4)良好的结构稳定性。基于第四代裂变反应堆更加苛刻的服役环境，传统的反应堆结构材料在相应的条件下服役，其高温力学性能（抗蠕变，高温持久性能）以及耐蚀性及抗辐照能力均不能满足需求。氧化物弥散强化(Oxide Dispersion strengthened, ODS)钢被列为多种裂变反应堆包壳及结构材料的候选材料，具有广阔的应用前景。

表 1 第四代裂变反应堆的服役条件及候选材料^[4, 5]

Table1 Service Conditions and Candidate Materials of the Fourth Generation Fission Reactor^[4, 5]

堆型	压力 (MPa)	温度 (°C)	能谱, 剂量 (dpa)	结构材料		
				包壳材料	芯内	芯外
钠冷堆	0.1	370~550	快, 200	F/M, F/M ODS	F/M,316SS	铁素体, 马氏体, F/M, 陶瓷, 难熔金

					属
液态铅铋堆	0.1	600~800	块, 150	F/M, ODS, 陶瓷, 难熔金属陶瓷	F/M, 陶瓷, 难熔金属
气冷反应堆	7	450~800	块, 80	陶瓷	难熔金属, 陶瓷, ODS, F/M 镍基合金等
熔盐堆	0.1	700~1000	热, 200	-	难熔金属, 镍基合金, 陶瓷, 石墨, 镍基合金
超高温反应堆	7	600~1000	热, <20	SiC, ZrC	石墨, SiC, ZrC, F/M 镍基合金, 低合金钢
超临界水反应堆	25	290~600	热, 30 快, 70	F/M, ODS	F/M, ODS 等 F/M, 低合金钢

2. ODS 钢及其研究进展

2.1 ODS 钢显微组织特点

ODS 钢在核能领域的研发始于上个世纪 60 年代，细小的晶粒尺寸、高数密度的位错及弥散分布的纳米氧化物粒子是性能优异的 ODS 钢典型的组织特征^[8-12]。其中晶粒尺寸一般为亚微米级，氧化物粒子尺寸仅为几个纳米，其所占的体积分数约为 0.5%，位错密度更是高达 10^{13} - 10^{15}m^{-2} ^[13]。图 2 为 14Cr-ODS 钢的晶粒形貌及析出相的 TEM 图片，平均晶粒尺寸为 200nm，析出相的尺寸约为 5nm^[14]。研究表明，与面心立方的奥氏体钢相比，体心立方结构的铁素体钢和铁素体/马氏体钢有着更加优异的抗辐照肿胀性能，因而 ODS 铁素体钢和 ODS 铁素体/马氏体钢成为当下研究的热点。

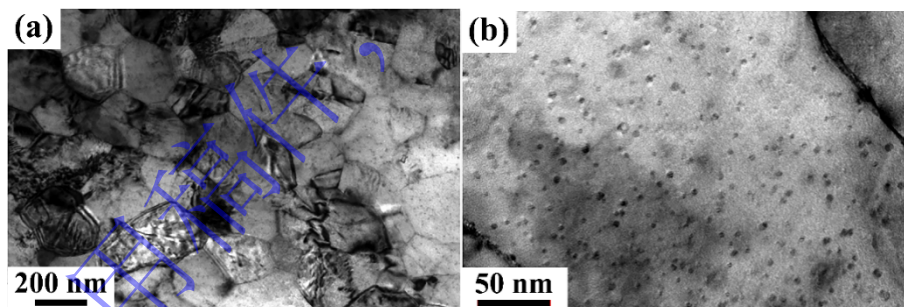


图 2 14Cr ODS 钢显微组织及析出相 TEM 图片^[14]

Fig.2 TEM pictures of microstructure and precipitates for 14Cr ODS steel^[14]

2.2 ODS 钢的强韧化机制

ODS 钢的常见的强化机制有如下四种：固溶强化(σ_m)，细晶强化(σ_k)，位错强化(σ_p)以及第二相强化(ρ_{dhh})。Kim 通过对 14YWT 不同温度下的力学性能表明，上述四种强化机制对强度的作用效果可用公式 2-1 来表达，可用于预测 ODS 钢的高温强度^[15]。

$$YS = \left[\sigma_p^2 + (\sigma_k^2 + \sigma_m^2 + \sigma_{dhh}^2)^2 \right]^{1/2}; \quad (2-1)$$

而上述的四种强化机制中，第二相强化对性能的提升贡献最大。典型代表强化机制为奥罗万强化机制，示意图如图 3 所示。

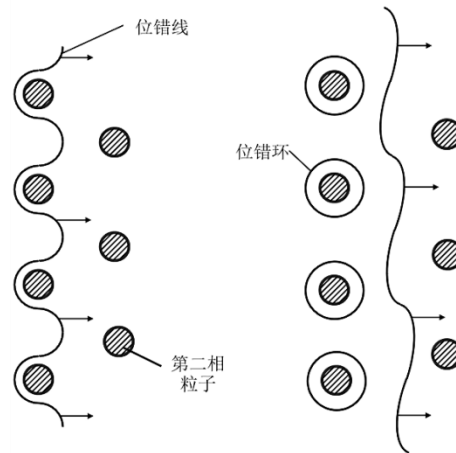


图3 奥罗万强化机制示意图

Fig.3 Schematic diagram of Orowan strengthening mechanism

位错线在运动过程中遇到第二相粒子会受到阻碍而发生弯曲环绕，随着外界载荷的增强，位错线会进一步向前运动而在第二相粒子留下位错环而使得位错可以进一步向前运动。对于 ODS 钢而言，由于其粒子的尺寸非常细小，且与基体的界面多为共格或半共格界面。而奥罗万强化机制对屈服强度的影响^[16, 17]，可表达为如下公式：

$$\sigma_{OR} = \frac{2m\mu b}{4\pi \times (1.18) \times (\lambda - \Phi)} \ln\left(\frac{\Phi}{2b}\right)$$

其中 σ_{OR} 代表该机制对屈服强度的贡献值， m 为泰勒因子， μ 为剪切模量， b 为伯氏矢量， Φ 为第二相粒子的尺寸， λ 代表的是粒子之间的间距。无疑在第二相粒子体积分数一定的情况下，粒子间距越小，粒子的数量越多，则材料的屈服强度越大。

高数密度弥散分布的氧化物粒子不仅可改善合金的强度，对材料的韧性也有很强的改善作用^[18-20]。细小的第二相粒子利于降低粒子/基体之间的应力集中程度，延缓了界面裂纹的萌生；晶粒内部第二相粒子的可将变形过程中产生的大量位错钉扎在晶内，抑制位错过早的运动到晶界处，合金在断裂前可承受更大的变形。

综合而言，为了使得 ODS 钢获得良好的综合强韧性，氧化物粒子须得满足以下要求：(1)粒子尺寸尽可能小，(2)微粒的间距须达到最佳程度；(3)微粒在基体中尽可能均匀分布；(4)微粒具有良好的热稳定性，高温下聚集长大的倾向性要小；(5)微粒不与基体发生反应。

2.3 ODS 钢的抗辐照机制

材料在高能快中子的辐照下，会产生大量的空位及间隙原子等缺陷，导致了材料的屈服强度上升、体积肿胀等问题，影响了材料的结构稳定性^[22-25]。而 ODS 钢自身存在的高数密度的弥散粒子-基体之间的界面可大幅度的改善材料的抗辐照性能^[26-28]。图 4 为 ODS 钢和传统商用钢在抗辐照性能上存在差异的示意图^[13]。ODS 钢中高数密度的氧化物粒子能够俘获辐照过程中产生的空位缺陷和氦泡，对缺陷的迁移和聚集起到很好的抑制作用，从而抑制了材料的辐照肿胀行为。而传统商用钢中因缺陷(主要指第二相)的数密度不足，难以抑制空位缺陷及氦泡的聚集长大，因而其抗辐照性能远低于 ODS 钢。

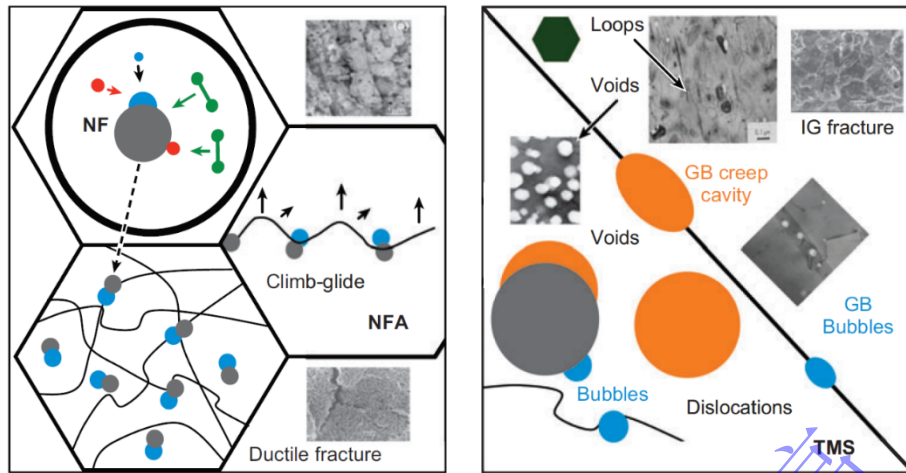


图 4 传统商用钢与 ODS 铁素体钢在抗辐照性能上存在差异的原因^[13]

Fig.4 Schematic of the difference in radiation resistance between traditional steels and ODS steels^[13]

3. ODS 钢的成分设计

3.1 典型的成分设计

基于先进核能系统苛刻的工况环境，世界各国围绕材料的性能目标要求针对 ODS 钢的成分设计及工艺制备开展了大量的研究工作。表 2 汇总统计了世界各国的典型核用 ODS 钢的成分。基于体心立方结构钢在抗辐照性能上的巨大优势，国外研究的重点多集中在研究重点多集中在 ODS F/M 钢和 ODS 铁素体钢，

表 2 典型核用 ODS 钢的成分（质量分数，wt%）

Table 2 Chemical composition of typical nuclear-grade ODS steels (mass fraction, %)

国家	牌号	Cr	W	Ti	Y ₂ O ₃	Other	Fe
美国	MA957 ^[29-34]	14		1	0.25	0.3Mo	Bal
美国	14YWT ^[35-39]	14	3	0.4	0.3		Bal
美国	OFRAC ^[40, 41]	12		0.3	0.3	0.3Nb+1Mo	Bal
美国	CRAZY ^[42]	10			0.3	0.06C+0.3Zr+6Al	Bal
日本	9Cr-ODS ^[43]	9	2	0.2	0.356	0.13C	Bal
日本	12Cr-ODS ^[44]	12	2	0.3	0.25	0.04	Bal
法国	9Cr-ODS ^[45-48]	9	1	0.2	0.3	0.1C	Bal
法国	14Cr-ODS ^[49-51]	14	1	0.3	0.3		Bal

由表 2 可知，Cr、W 和 Mo 是 ODS 钢主要的固溶强化元素。Cr 的添加量通常在 9~14wt% 之间，其含量变化对显微组织及拉伸性能无明显影响^[52]。Cr 含量大于 14wt% 利于稳定铁素体组织的形成，并且可显著提高合金的抗高温氧化性能。但高 Cr 钢在 300~550℃ 长期服役的过程中会有 α' 富 Cr 相的析出，这会导致时效脆化问题的产生，故而 Cr 的添加量一般不超过 14wt%^[53-56]。W 和 Mo 的添加可促进稳定铁素体组织的形成并改善合金的高温强度。但过量的 W 或者 Mo 的添加会导致长时时效过程中晶界处片层状 Laves 相的析出从而降低到合金的塑韧性。通常 W 和 Mo 的总添加量一般不超过 3wt%。

含 Al ODS 钢是近些年来的研究热点之一，Al 的添加可极大的改善合金的抗氧化性能及耐蚀性能^[57, 58]。研究表明：16Cr-ODS^[59] 钢中，Al 的添加量大于 3wt% 时可在合金表面形成比 Cr₂O₃ 更加稳定、致密 Al₂O₃ 膜。并且由“第三元素效应” (The third-element effect)^[60]，Cr 会促进致密 Al₂O₃ 氧化膜的形成^[61]。研究表明：APM 与 Zr-4 在 1200℃ 蒸汽环境下的质

量增长对比图, APM 的质量增重($60\text{mg}/\text{dm}^2$)仅为 Zr-4($3200\text{mg}/\text{dm}^2$)的 $1/50$ ^[62]。然而, 商用 ODS-FeCrAl 合金(APM, APMT)在液态金属钠 $700^\circ\text{C}/1000\text{h}$ 的静态腐蚀行为表明: FeCrAl 合金中的 Al 会与 O、Na 优先发生反应, 生成 Na-Al-O 的三元氧化物并溶解于液态金属钠中^[63], 加速了材料的腐蚀行为。故通过添加 Al 来改善燃料包壳材料与冷却介质的兼容性并不是一个通用的手段, 还需针对不同冷却介质开展针对性的研发工作。

3.2 微合金元素的影响

ODS 钢中添加微量元素主要是为了与 O 元素等发生反应, 在合金内部生成高数密度的纳米氧化物粒子^[42, 64-67]。大量的研究表明, 微合金元素的复合添加对弥散颗粒的类型、尺寸及分布及稳定性有着重要的影响, 进而影响材料的部分服役性能(高温持久性能、抗辐照性能)等^[68-74]。表 3 汇总统计了 ODS 钢中的不同类型的氧化物粒子的成分、结构等相关信息。由表 3 可知, 含 Y 三元氧化物复合粒子的标准生成焓均低于 Y_2O_3 , 有着比 Y_2O_3 更高的热力学稳定性, 并且实际的研究表明三元氧化物粒子一般小出 Y_2O_3 约一个数量级以上, 多集中在 30nm 以下^[75]。而在含 Al ODS 钢中, 因 Al 元素有着较高的亲氧性, 合金中会优先析出 Y-Al-O 三元氧化物粒子^[76, 77]。多数研究发现, 含 Al 氧化物粒子的尺寸通常相对较大且易粗化, 故而在含 Al ODS 钢中会选择添加 Zr, Si 等元素, 在合金中优先析出尺寸相对较小的 Y-X(X=Zr, Si)-O 复合氧化物粒子, 从而改善氧化物粒子的尺寸^[68, 69, 78-81]。

表 3 ODS 钢中不同类型含 Y 氧化物粒子基本信息

Table 3 Basic information of different types of Y-containing oxide particles in ODS steel

合金元素	氧化物类型	标准生成焓	晶体结构	形成机理
Ti	Y_2O_3	-1905 kJ/mol ^[82]	Cubic	$4\text{Y}+3\text{O}_2\rightarrow 2\text{Y}_2\text{O}_3$ ^[83]
	Y_2TiO_5	-2674 kJ/mol ^[84]	Orthorhombic	$\text{Y}_2\text{O}_3+\text{TiO}_2\rightarrow \text{Y}_2\text{TiO}_5$ ^[83]
	$\text{Y}_2\text{Ti}_2\text{O}_7$	-3874 kJ/mol ^[71]	Cubic	$\text{Y}_2\text{O}_3+2\text{TiO}_2\rightarrow \text{Y}_2\text{Ti}_2\text{O}_7$ ^[69]
Al	$\text{Y}_4\text{Al}_2\text{O}_9$	-5546 kJ/mol ^[85]	Monoclinic	$2\text{Y}_2\text{O}_3+\text{Al}_2\text{O}_3\rightarrow \text{Y}_4\text{Al}_2\text{O}_9$ ^[69, 85]
	$\text{Y}_3\text{Al}_5\text{O}_{12}$	-7197 kJ/mol ^[85]	Cubic	$3\text{Y}_2\text{O}_3+5\text{Al}_2\text{O}_3\rightarrow 2\text{Y}_3\text{Al}_5\text{O}_{12}$ ^[69]
Zr	YAlO_3	-1827 kJ/mol ^[85]	Hexagonal	$\text{Y}_2\text{O}_3+\text{Al}_2\text{O}_3\rightarrow 2\text{YAlO}_3$ ^[69, 85]
	$\text{Y}_4\text{Zr}_3\text{O}_{12}$	-	Trigonal	$2\text{Y}_2\text{O}_3+3\text{ZrO}_2\rightarrow \text{Y}_4\text{Zr}_3\text{O}_{12}$ ^[69, 85]
	$\text{Y}_2\text{Zr}_2\text{O}_7$	-	Cubic	$\text{Y}_2\text{O}_3+2\text{ZrO}_2\rightarrow \text{Y}_2\text{Zr}_2\text{O}_7$ ^[69]
Si	Y_2SiO_5	-2869 kJ/mol ^[84]	Monoclinic	$\text{Y}_2\text{O}_3+\text{SiO}_2\rightarrow \text{Y}_2\text{SiO}_5$ ^[86]
	$\text{Y}_2\text{Si}_2\text{O}_7$	-3820 kJ/mol ^[87]	Monoclinic	$\text{Y}_2\text{O}_3+2\text{SiO}_2\rightarrow \text{Y}_2\text{Si}_2\text{O}_7$ ^[86, 88]

除元素类型会影响到氧化物的类型及尺寸, 元素之间的配比也会影响到氧化物的类型及尺寸^[89, 90]。图 5^[32]展示了 MA957 中 Y/Ti 比与弥散粒尺寸的关系。Y-Ti-O 氧化物可按是否符合化学计量比分为两大类。符合化学计量比(Y/Ti \approx 1)的氧化物主要包括 Y_2TiO_5 ^[91-93]和 $\text{Y}_2\text{Ti}_2\text{O}_7$ ^[91-93], 这些颗粒粒径在 $15\sim 35\text{nm}$ 之间。另一类则不符合化学计量比, 这一类弥散氧化物颗粒粒径在 $2\sim 5\text{nm}$ 之间, 多以纳米团簇形式出现。

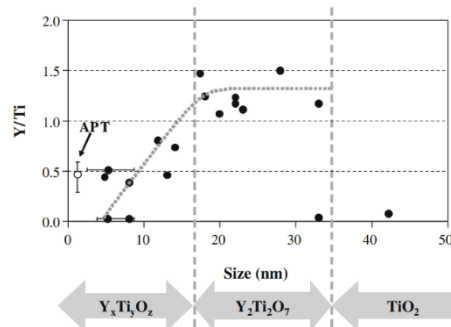


图 5 MA957 中 Y/Ti 比值与析出相尺寸的关系^[30]

Fig.5 Relationship between Y/Ti ratio and dispersed particle size in MA957^[30]

4. 制备工艺

4.1 机械合金化

合金中氧化物粒子的形状、尺寸和分布对于 ODS 钢的性能至关重要。为能够在基体内析出细小弥散均匀分布的氧化物颗粒，通常采用机械合金化(Mechanical Alloying, MA)-粉末冶金(Powder Metallurgy, PM)工艺来制备 ODS 钢^[92]。MA-PM 制备 ODS 钢的工艺流程图^[13]如图 6 所示，包括：气雾化制粉，机械合金化(Mechanical alloying, MA)，脱气封装，热等静压及后续热变形处理^[11]。

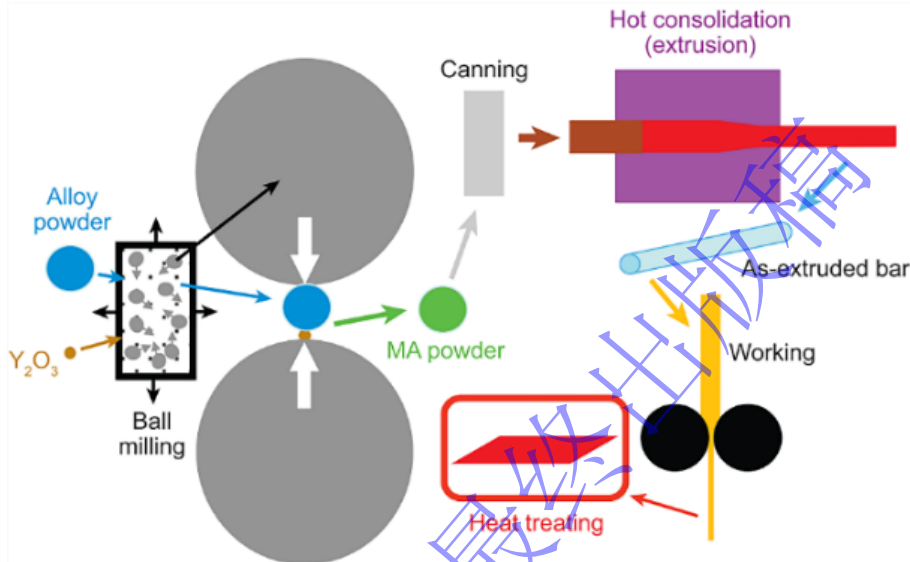


图 6 ODS 钢制备工艺流程图^[13]

Fig.6 Flow chart of preparation process for ODS steel^[13]

MA 是指将预合金粉末和 Y₂O₃ 粉末颗粒混合并进行高能球磨，金属粉末经过长时间冲击碰撞，自身不断的冷焊、断裂和冷焊，粉末的组织结构不断的细化，最终达到原子级别的混合状态^[95]。研究表明，机械合金化过程可以使得 Y 和 O 过饱和和固溶于合金粉末当中^[96]。高温烧结过程中粉末中过饱和的 Y 和 O 会部分析出并与粉末中的 Ti、Al 等元素形成稳定、弥散分布的 Y-X-O(X=Ti、Zr、Al、Hf 等)的团簇或者氧化物粒子^[37, 89, 97-105]，从而达到均匀弥散分布的效果。

尽管 MA+HIP 是目前制备 ODS 钢最主要的制备工艺，但是该工艺存在一些固有缺点。工艺流程耗时长、单批次产量有限。为制备出符合预期的预合金化粉末，球磨时间可高达 50h^[81]。其次成分的均质性难以控制，粉末容易被污染和氧化。高能球磨过程中所使用的高碳钢磨球会对粉末造成 C 污染，粉末经过球磨处理后其碳含量从初始的 ppm 最高增加至 1200ppm^[106]。而碳污染则会诱发 M₂₃C₆ 等富 Cr 碳化物的形成，同时容易引起材料局部贫 Cr 现象，会恶化力学性能及耐蚀性能^[107]。最重要的一点就是低效，考虑到行星式球磨机的罐体每批次罐体的装粉量有限，严重限制了其在工业生产中的大规模应用。因而急需寻求可替代工艺，提高 ODS 钢的制备效率。

4.2 粉末氧化法

粉末氧化法是根据不同氧化物生成焓之间的差异，通过调整制粉工艺或者对粉末进行后处理，在粉末表面形成一定的厚度的亚稳态氧化层。在高温烧结的过程中，粉末表层的亚稳态氧化物发生分解，与粉末内部的 Y、Ti 等活性元素发生反应，在合金内部析出热力学更加稳定的氧化物粒子。Pazos 等人^[108-110]利用活性气体雾化方法制备表面含 Cr 亚稳氧化薄层的金属粉末，富 Cr 氧化层作为中间载体在高温烧结过程中发生分解并为含 Y 和 Ti 的氧化物粒子的形成提供氧元素，利用此方法制备了含氧化物粒子的合金。与 MA 方法相

比，粉末氧化法制备 ODS 钢省去了长时低效的 MA 过程，并且单批次粉末处理量大，极大的缩短了制备周期，极大的提高 ODS 钢的制备效率。但是与 MA-PM ODS 钢相比，性能还存在有很大的差距，原因在于雾化制粉过程中微量元素分布均匀性的控制与细粉收得率有待提高^[109]。且该工艺方法应用在含 Al ODS 钢的制备上存在一定的困难，Al 会与环境介质中的 O₂ 发生反应并在粉末表面迅速形成致密的 Al₂O₃ 膜，阻碍氧化过程的进一步进行并影响富 Al 和 Y 的氧化物弥散相的形成^[75]。

4.3 增材制造技术

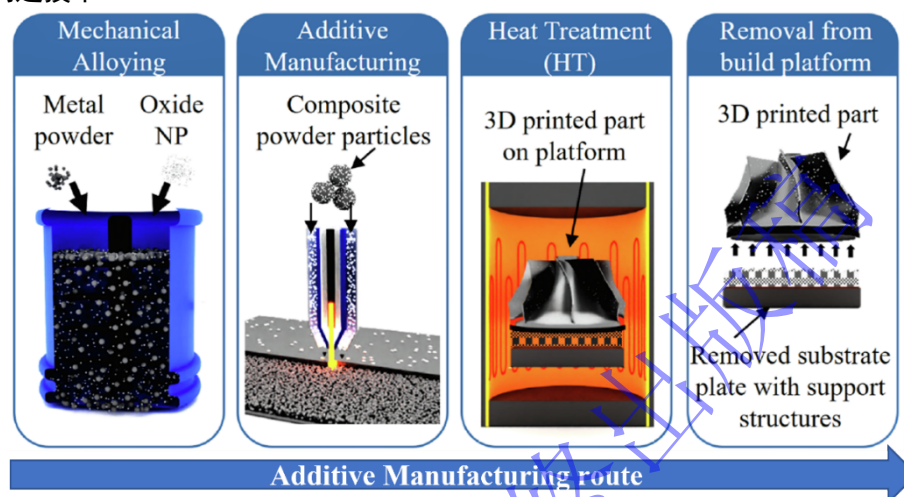


图 7 ODS 钢的增材制造工艺流程图^[111]

Fig.7 Flow chart of additive manufacturing process for ODS steel^[111]

基于粉末的 3D 打印技术也是一种很有前景的 ODS 钢制备技术，如图 7^[111]所示，该技术路径能够实现从粉末到产品的近净成型，直接省去了变形加工环节，缩短了制备周期。图 8^[112]为增材制造技术制备的 14Cr ODS 钢及其显微组织和纳米氧化物粒子的尺寸统计图。结果表明：MA 处理后的合金粉末在经过激光束融化后，合金内部可形成一定数密度的纳米氧化物粒子。但是总体而言，粉末融化过程的存在不可避免的导致了晶粒的粗化以及氧化物粒子的偏聚及长大^[112-115]。除此之外，利用该技术大规模制备高性能 ODS 钢仍面临许多挑战，包括适用于增材制造的粉末原料的大规模制备方法等。

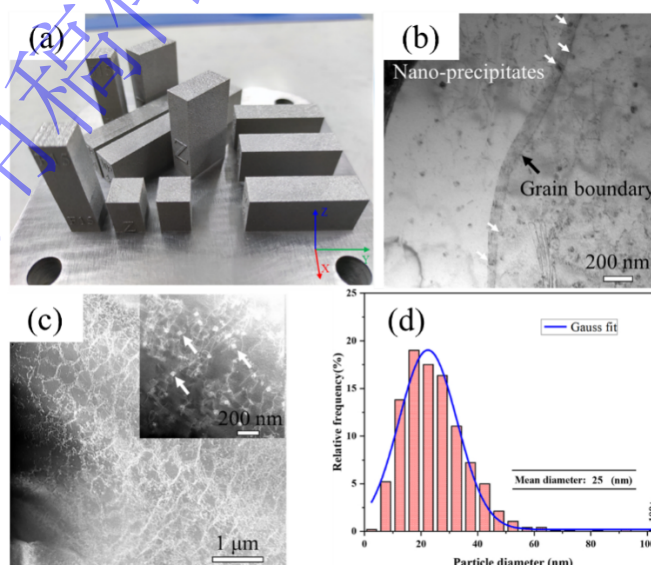


图 8 增材制造技术制备的 ODS 钢及其显微组织和纳米氧化物粒子尺寸统计图^[112]

Fig.8 Statistical diagram of ODS steel and its microstructure and size of nano-oxide particles

prepared by additive manufacturing^[112]

4.4 放电等离子烧结技术

放电等离子烧结是指在真空环境下利用单向压力和脉冲直流电流使合金粉末快速热固结的技术^[116-119]，图 9 为放电等离子烧结设备示意图^[113]。作为一种新型的热固化工艺，该烧结工艺有如下优点：(1)烧结速度快，可有效防止晶粒过度长大；(2)烧结温度相对较低，比 HIP 低 200~300℃左右；(3)制备出的样品无明显的各向异性。目前越来越多的研究人员将该技术应用于 ODS 钢的制备。

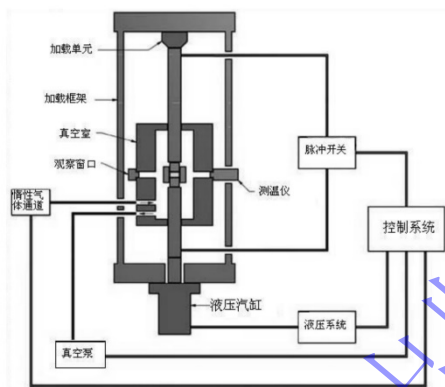


图 9 放电等离子烧结设备示意图^[116]

Fig.9 Schematic representation of a spark plasma sintering system^[116]

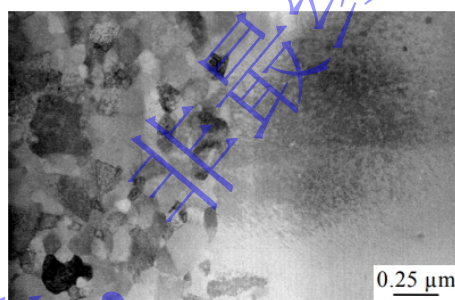


图 10 SPS 工艺制备而成的 9Cr-ODS 钢的显微组织^[120]

Fig.10 Microstructure of 9Cr-ODS steel produced via SPS^[120]

图 10 展示了放电等离子烧结工艺制备而成的 9Cr ODS 钢的显微组织，晶粒呈双峰分布，这与烧结机制有关，烧结中由于粉末颗粒表面的电阻小电流主要沿着粉末颗粒表面运动，因而粉末颗粒表面被快速加热冷却，有着比粉末内部相对较高的温度，颗粒容易粗化，形成大晶粒区，颗粒内部相对温度较低，晶粒长大速率较慢，形成超细晶粒区^[120]。

但该技术在推广应用的过程中仍存在一些挑战：(1)烧结体尺寸偏小；(2)产品成本偏高，石墨磨具损耗较大，整体寿命较短，增加了产品的生产成本；(3)磨具污染，石墨磨具在使用过程中存在碳原子向烧结体扩散，导致烧结体表面甚至内部扩散发生碳污染^[116]。

4.5 络合溶胶-凝胶法

络合溶胶-凝胶法是一种前驱体粉末制备技术。以乙二胺四乙酸(EDTA)为络合剂，将气雾化预合金粉末和硝酸钇分别按照一定的比例添加，随后水浴加热并烘干处理，从而在粉末表层形成分散性较好的纳米 Y_2O_3 粒子^[121]。

图 11 展示了利用络合溶胶-凝胶法+热等静压+锻造制备而成的 12Cr-ODS 钢显微组织图片以及拉伸性能^[122]。由图可知，合金中形成有高数密度的氧化物粒子，多数氧化物粒子的尺寸在 100nm 以下。实验结果表明，该材料在高温下仍具有较高的强度，在 550℃温度条件下，其抗拉强度不低于 1000MPa，并且具有相对较低的韧脆转变温度-25℃^[122]。

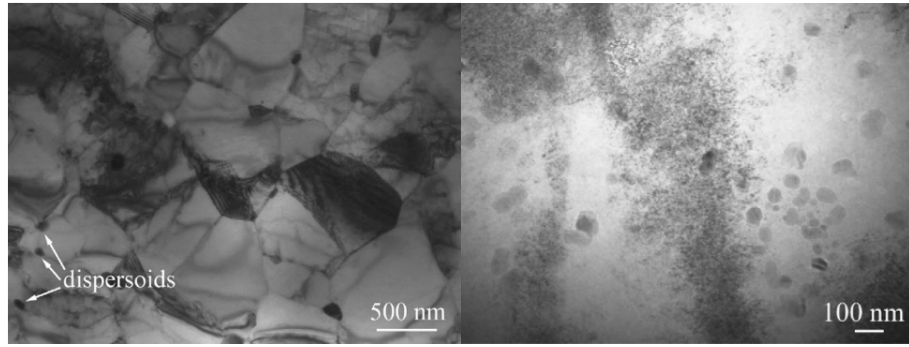


图 11 络合溶胶-凝胶法工艺路径制备而成的 12Cr-ODS 钢的 TEM 图片^[122]

Fig.11 TEM pictures of 12Cr-ODS steel fabricated through complex sol-gel method^[122]

4.6 粉末爆炸压实技术

爆炸压实是利用炸药爆炸时产生的冲击波把金属或非金属粉末压实烧结成形的加工工艺^[123]，有着广阔的应用前景。在对粉末爆炸压实的过程中，爆炸产生的冲击波能够破碎粉末表层的亚稳态样层，且粉末在爆炸压实过程中产生的大量缺陷成为氧化物粒子析出的优先形核点，利于氧化物粒子在后续的热处理过程中均匀析出^[123, 124]。已有研究表明：爆炸压实能够使粉末实现致密化成型，坯料的致密度高达 98%；并且压坯可以在相对较低的温度下进行热处理，避免了成型温度过高导致的氧化物粒子的粗化^[124]。与热等静压烧结相比，该工艺方法将致密化环节与氧化物高温析出环节实现了分离，可通过调节压坯的热处理时间与温度来实现对氧化物粒子尺寸的调控；但过程环节的增加同时也增加了材料的制备周期。

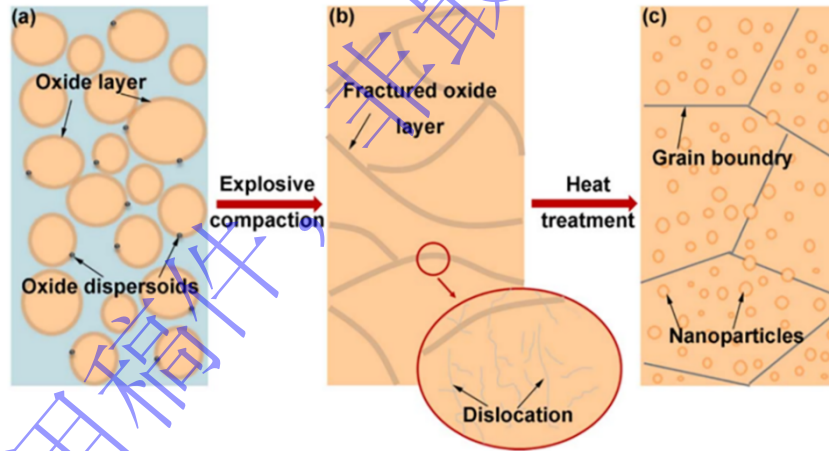


图 12 氧化粉末爆炸压实及析出相形成示意图^[124]

Fig.12 Illustration for the formation of precipitates in ODS alloys by direct oxidation and explosive compaction^[124]

4.7 粉末锻造技术

粉末锻造成型工艺是传统的粉末冶金工艺与锻造结合而形成的一种新型成型工艺^[125,126]。该工艺涉及粉末预压、加热、保温及锻造成型等工序。粉末锻造成型后合金内部组织均匀，无明显偏析消除了常规锻造材料的各向异性；粉末在锻造过程中会产生大量的位错、空位等缺陷，利于高数密度纳米氧化物弥散相的析出。并且，粉末锻造成型具有工序少、流程短、效率高等优点^[121]。

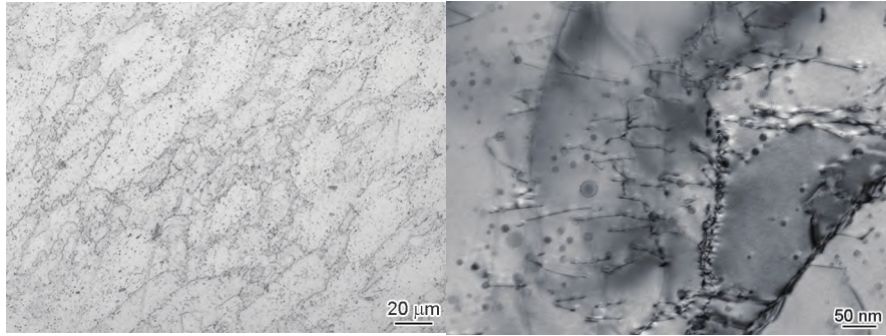


图 13 粉锻成型后 ODS-FeCr 合金的光镜图片(左)和透射图片(右)^[127]

Fig.13 OM (left) and TEM picture(right) of a 9Cr-ODS steel via powder forging process^[127]

图 13 为粉末锻造处理后的 ODS-FeCr 合金的光镜图片和 TEM 图片，合金内部结构致密，无明显的孔洞、裂纹，PPB 等缺陷，在合金内部观测到有高数密度的氧化物粒子和位错；其致密度高达 99.5%^[127]。

4.8 熔炼铸造技术

熔炼铸造法制备 ODS 钢是将含 Y 钢液浇筑至预先铺好 Fe_2O_3 粉末的模具中，浇筑过程中 Fe_2O_3 中的 O 会与钢液中的 Y 发生反应原位生成含 Y 纳米氧化物粒子，其流程示意图如图 14(a)所示^[128]。与粉末冶金工艺相比，熔炼铸造法制备 ODS 钢具有工艺流程短、单批次产量大等优点。但在熔炼法制备 ODS 钢的过程中仍存在一些挑战，即抑制氧化物粒子的团聚和粗化^[128, 129]。

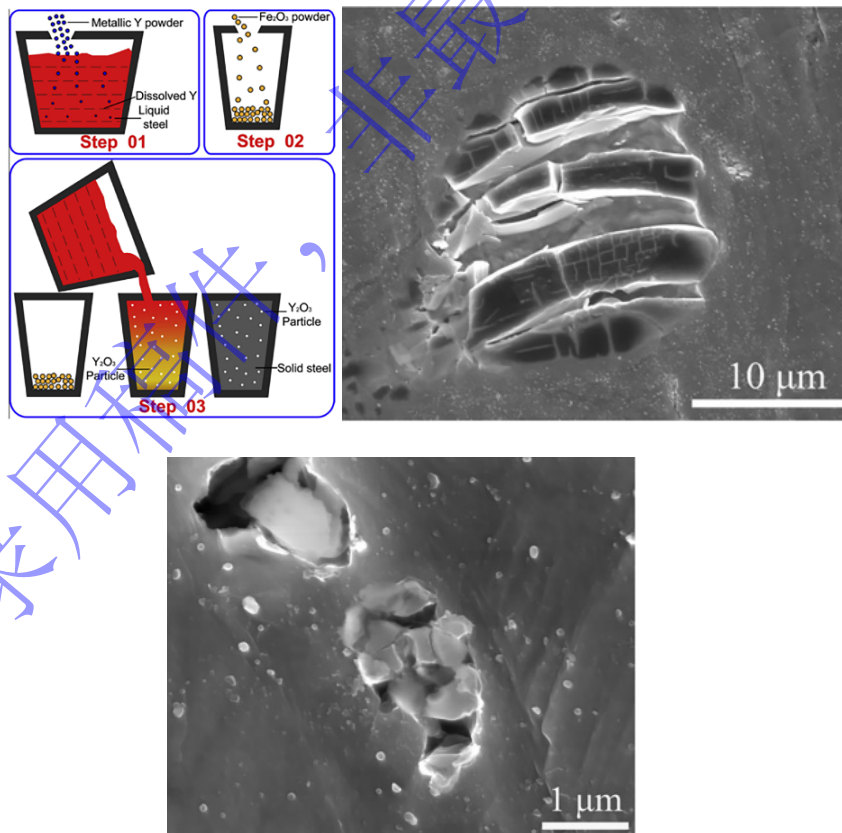


图 14 熔炼铸造法制备 ODS 钢示意图^[128]及含 Y 氧化物粒子的粗化与偏聚^[129]

Fig.14 Schematic diagram of ODS steel preparation by smelting and casting method ^[128] and coarsening and segregation of Y-containing oxide particles ^[129]

5. 结语及展望

先进核能系统苛刻的服役环境对材料的综合服役性能有着更高的要求。在钢中引入高数密度弥散分布的氧化物粒子可大幅度改善并提高材料的组织稳定性、高温性能和抗辐照性能，使得 ODS 钢能够满足核能苛刻环境下对材料的性能要求。因而 ODS 钢成为第四代核能系统反应堆包壳重要的候选材料。

综合已有研究成果发现，体心立方结构的 ODS 钢（铁素体钢，F/M 钢）有望第四代核能系统中获得应用。目前现阶段在研 ODS 钢或多或少都存在一些问题：高 Cr 钢易时效脆化、含 Al 粒子容易粗化、材料与部分冷却介质之间的存在兼容性不匹配等；这就需要综合前人已有工作的基础上，针对不同的反应堆型，开展相应的成分优化设计工作，实现组织及性能的综合提高。

材料的高效批量制备技术是 ODS 钢走向工程应用的首要环节。传统的 MA-PM 制备技术因存在低效、均质性差等问题限制了其在工业生产中的应用。而 ODS 钢改进型制备工艺，均可缩短材料的制备周期并提高了生产效率，表现出极大的应用潜力。但是在组织与性能上与 MA-ODS 钢相比存在一定的差距。这需要针对各自的制备过程开展相应的优化工作。以为粉末为原料的粉末氧化法、增材制造技术以及粉末爆炸压实技术等，需要开展粉末制备工艺优化工作，优化粉末粒度并提高微量元素在粉末中的均匀分布，同时先驱体粉末中的氧含量的调控亦是值得研究的重点。熔炼铸造法可试制出含有纳米氧化物粒子 ODS 钢，但是需重点解决制备过程中出现的氧化物团聚与粗化。

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