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Review on the EFDA programme on tungsten materials technology and science

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ABSTRACT

All the recent DEMO design studies for helium cooled divertors utilize tungsten materials and alloys, mainly due to their high temperature strength, good thermal conductivity, low erosion, and comparably low activation under neutron irradiation. The long-term objective of the EFDA fusion materials programme is to develop structural as well as armor materials in combination with the necessary production and fabrication technologies for future divertor concepts. The programmatic roadmap is structured into four engineering research lines which comprise fabrication process development, structural material development, armor material optimization, and irradiation performance testing, which are complemented by a fundamental research programme on "Materials Science and Modeling". This paper presents the current research status of the EFDA experimental and testing investigations, and gives a detailed overview of the latest results on fabrication, joining, high heat flux testing, plasticity, modeling, and validation experiments.

1. Introduction

Tungsten and tungsten alloys are presently considered as candidate materials for the helium cooled divertor and possibly for the protection of the helium cooled first wall in DEMO designs, mainly because of their high temperature strength, good thermal conductivity, and low sputter rates [1–5]. There are two types of

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applications for these materials which require quite different properties: one is for their use as plasma-facing armor or shield component, the other is for structural purposes. An armor material needs high crack resistance under extreme thermal operation conditions [6,7] and compatibility with plasma–wall interaction phenomena [8], while a structural material has to be ductile within the operation temperature range. Both material types have also to be stable with respect to high neutron irradiation doses and helium production rates.

The long-term objective of the present EFDA programme is to provide structural as well as armor materials in combination with the necessary production and fabrication technologies for future divertor components. On a European level, fusion materials research is strengthened by integrating new partners, in particular large-scale facilities like synchrotron and neutron laboratories, into the materials development and characterization process by the FP7 coordination action “FEMaS” [9]. Presently there are many unsolved issues, contradictions, and problems related to the use and properties of tungsten materials. Therefore the roadmap is structured into four lines of classical engineering research: (1) Fabrication process development, (2) structural material development, (3) armor material optimization, and (4) irradiation performance testing. They are complemented by an additional basic research line: (5) Materials science and modeling.

Irradiation performance testing provides experimental information on neutron irradiation damage of tungsten and tungsten alloys. In the near future, this area will be kept at a low level, since all the presently available tungsten grades, which have been irradiated, show extreme post-irradiation brittleness. Also, this EFDA programme and the cooperation with external irradiation programs (e.g. during the ExtreMat project [10]) will give new directions for improvement, which will have to be formulated into a metallurgical specification to fabricate improved W-based materials and/or W-alloys for functional and structural applications.

However, the main objective of all research areas is the identification of applicability restrictions for tungsten and tungsten-based materials for their use as helium cooled divertor parts. This will be performed in the short- and mid-term program so that by the end of 2012 possible show stoppers will be identified and possible alternative solutions might be presented, if necessary.

To make the programme as economic as possible, the different research areas are also interlinked to other EFDA and external groups with respect to information and materials exchange.

2. Fabrication process development

The current helium cooled finger design [3] works in principle with pure W and W–La_2O_3 and relies on standard fabrication methods like turning from full rod material and standard brazing materials like, for example, copper. Therefore, a significant improvement with respect to quality, costs, and material selection is needed. Typical important questions in this field are: How to avoid micro-cracks? What alternative fabrication process could be suitable? Are there applicable reduced activation brazing materials for W–W and W–steel joints? Can mass production processes be applied to tungsten parts?

In summary, the long-term objectives are (1) to identify, qualify, and provide all fabrication steps and processing parameters necessary for the divertor part assembly and (2) to verify the reliability and lifetime of divertor parts by performing relevant component tests, i.e. tests in special helium loop facilities and tokamaks.

2.1. Machining

The main result of a comparative fabrication process investigation is that by turning and milling defect free surfaces can be produced (see Fig. 1). However, shaping of curved surfaces can be performed at a higher quality level by milling. Electrical discharge machining (EDM) often leads to surface cracks along the grain boundaries [11], while the use of diamond cutting or sawing produced no defects.

By adopting several electro-chemical machining (ECM) processes, further alternatives for crack-free fabrication of tungsten parts could be utilized. Surface electro-polishing was shown to remove cracks and grooves. It can also be applied to structures with high aspect ratios such as castellation gaps in plasma-facing armor tiles. An alternative process for structuring complex surfaces is the use of a movable cathode. By the proper choice of current density, step rate, pulse profile, electrolyte, and its convection even fine contours and relatively sharp edges can be fabricated [12].

Furthermore, the development of suitable mass production methods for divertor parts was started. Powder injection molding has been used to produce material test specimens and to demonstrate the feasibility [13]. Up to now, the extreme brittleness of such parts under dynamic loads is the main drawback of the method and has to be improved. But ductility measured by tensile tests

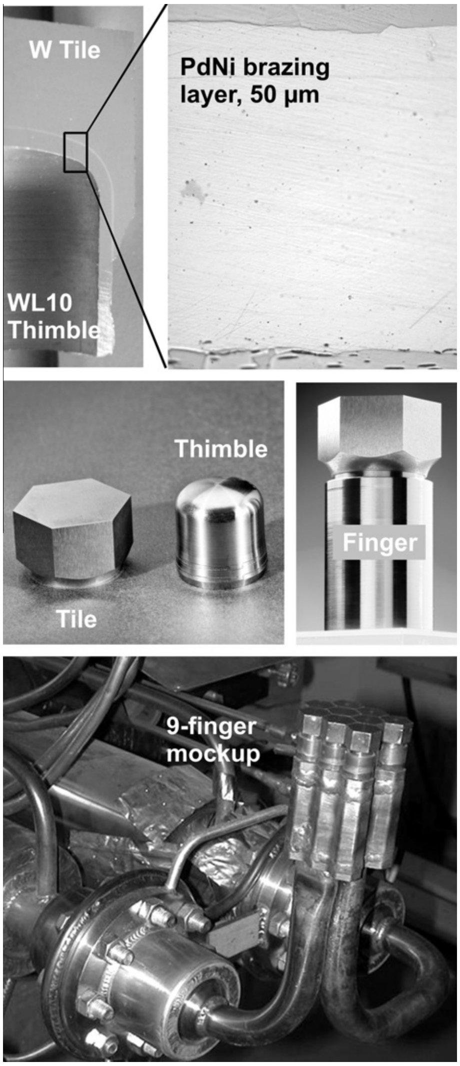

Fig. 1. An important fabrication step is joining tungsten parts. For the case of a jet-cooled finger the upper images show a successful PdNi brazing layer between a WL10 thimble and a W tile. Fabrication of the single parts has been optimized to produce smooth crack-free surfaces. Lower photo: A helium cooled nine-finger divertor module prepared for high heat flux test in the electron beam facility at Efremov Institute, Saint Petersburg, Russia. Courtesy of P. Norajitra, FZK, Germany.

is clearly better compared to commercial tungsten rod materials. High quality and net shape powder injection molding products are also developed by industry [14]. Press rolling of thimble-like parts were also studied. The process was setup and developed successfully for steel and TZM (molybdenum material). The applicability of the process to tungsten materials is yet to be proven. Deep drawing could be a further option for the fabrication of cooling parts.

2.2. Joining

Prior to the development and testing of low-activation brazing materials, a screening study of commercial brazes including interface behavior was performed and evaluated. Initially, for tungsten–tungsten joints the amorphous brazing foil STEMET 1311 was used at a brazing temperature of 1100 °C. But during mockup tests the joint often failed by detachments. In further steps a PdNi40 foil was used to optimize a joint between a WL10 thimble and a tungsten tile. Brazing temperatures around 1300 °C led to acceptable results (see Fig. 1). For a further increased brazing temperature CuNi44 could also be used. Future efforts have to concentrate on low activation materials. Therefore, one of the next steps is to investigate the feasibility of pure titanium as brazing material.

In principle brazing foils could be replaced by coating the joint surfaces. Different electro-deposition processes were investigated on the basis of aqueous and organic systems. The feasibility has been demonstrated by the deposition of a 200 μm thick Ni layer on tungsten. Of course, the method had to be adjusted to more complex brazing materials. By the use of ionic liquids, it was also possible to produce a 10 μm tungsten layer on Eurofer.

Alternative technologies like high power laser diode brazing and pulse plasma sintering were applied to TiCu40 foils (in two different conditions), to several experimental Ti–Fe powders, and to a commercial Ni23Mn7Si5Cu foil. Preliminary results are promising, but the brazing temperatures for Ti–Cu are probably too low to meet elevated operation temperature requirements.

The joints between tungsten and steel have to withstand the mismatch of the thermal expansion coefficient. Therefore, in initial tests the brazing has been performed by copper and later on by CuPd18 at temperatures around 1100 °C. But a low-activation brazing candidate material has still to be selected. In order to avoid the tungsten-to-steel brazing problem, an exploration and possible development of functional gradient materials have been started. The basic idea is to reach a local chemical equilibrium at any point in the transition by a two-step approach: First from tungsten to tungsten–carbide (WC) and then from WC to WC–Fe and Eurofer. First thermo-mechanical calculations show promising results.

2.3. Engineering testing

Several high heat flux test series on cooling finger mock-ups in the Efremov Institute, Saint Petersburg, Russia (electron beam facility combined with helium loop) have brought better insight into the complex load situation of divertor parts [11]. The feasibility of helium cooled divertor fingers was demonstrated by first mock-ups which survived 1000 cycles at 10 MW/m^2. Further optimized one-finger as well as nine-finger modules (see Fig. 1) were manufactured and assembled for testing by end of 2009.

3. Structural materials development

The goal is to find a ductile refractory material with acceptable thermal conductivity. So far the characterization of standard materials has shown that W–1%La$_2$O$_3$ (WL10) yields enough creep strength and heat conductivity for the present He cooled divertor design. But high DBTT values, combined with anisotropic microstructures, are the intrinsic characteristic of all available tungsten materials, which is still the main problem for their use as structural materials. Therefore, the remaining questions are: Can the DBTT be significantly decreased? Is it possible to reach a compromise between strength, ductility, and heat conductivity? Is it necessary to produce an isotropic microstructure?

3.1. Development and production

One of the possible candidate materials are W–Ta alloys. The present programme contains laboratory and industry scale production by powder mixing, pressing, sintering, and final cold/hot work. Microstructure analysis by electron backscatter diffraction (EBSD) of industrially produced batches showed a finer grain and somewhat larger misorientation in the case of W–5%Ta compared to W–1%Ta (see Fig. 2). Fracture toughness tests are currently prepared and will be performed in the near future.

Another production route consists of mechanical alloying (milling) or mixing, hot isostatic pressing (HIP), and eventually cold/hot work. Currently, the milling parameters are studied and optimized for W–10%Ta and W–20%Ta. The same fabrication route has already been applied to different mixtures of W, W–Ti, W–V, W–Y$_2$O$_3$, W–Ti–Y$_2$O$_3$, and W–V–Y$_2$O$_3$ powders. After blending and milling, the powders were canned, degassed, and sealed. Two final HIP processes consolidated the materials. Fracture toughness and strength are clearly increased for the W–Ti and W–Ti–Y$_2$O$_3$ alloys

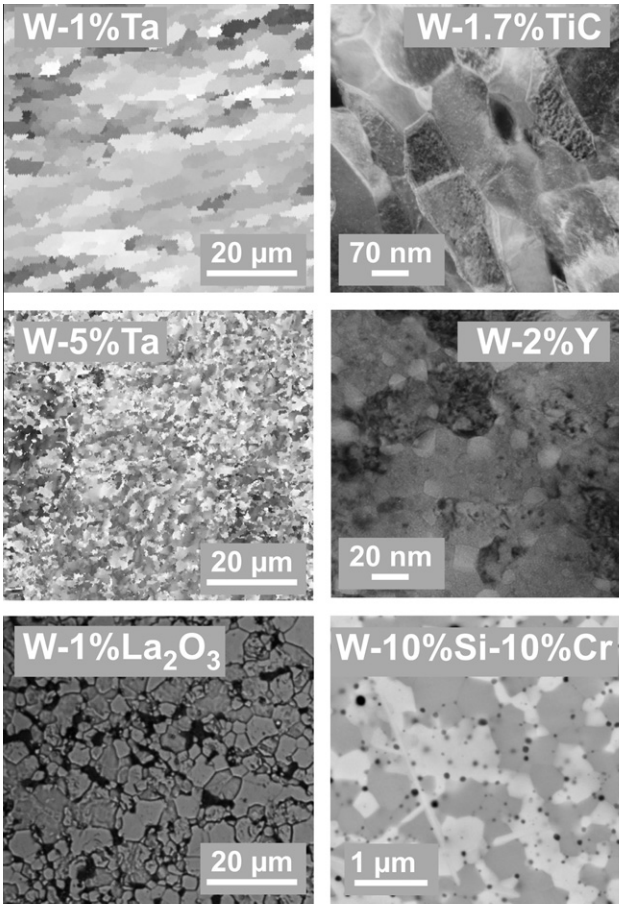

Fig. 2. Microstructure of industry fabricated W–1%Ta and W–5%Ta alloys compared to W–1%La$_2$O$_3$, W–2%Y, W–1.7%TiC, and W–Si–Cr materials fabricated by mechanical alloying on laboratory scale. Courtesy of R. Pippan, ÖAW, Austria, A. Muñoz, CIEMAT/UC3M, Spain, N. Baluc, CRPP, Swiss, and C. García-Rosales, CEIT, Spain.

compared to pure W. The characterization of the W–V alloys is still ongoing. However, the microstructure examinations reveal that there is still room for optimization of the production process (see Fig. 2).

The third method to produce nano-structured tungsten materials is based on chemical powder metallurgy. Starting from a precursor in aqueous solution powder processing is performed by calcinations and one or more reduction steps. For the final consolidation spark plasma sintering will be applied. Up to now only small quantities of W–Y_2O_3 and W–La_2O_3 have been produced. The ongoing work will also consider the W–V system and concentrate on the up-scaling of the whole fabrication process.

3.2. Characterization

For clear comparability all the produced materials have to be characterized by basic, standardized methods which are (1) DBTT measurement either by Charpy (KLST standard) or bending tests with different strain rates [15–17], (2) creep/tensile/indentation tests at 1100–1300 °C, (3) thermal conductivity measurements, (4) determination of re-crystallization temperature, and (5) microstructure and fracture analysis. Within the present EFDA programme, two large batches of the most promising (commercial grade) tungsten materials were ordered and characterized to establish a database for future recording of the progress in material development. Where necessary, the basic characterization programme is complemented by special tests like, for example, sub-miniaturized fracture mechanics or high temperature low-cycle fatigue.

4. Armor materials optimization

The aims of the material optimization program depend on the expected operation temperatures. The divertor plasma-facing material will be operated at temperatures up to 1700 °C or even more, whereas the operation temperature for the thermally lower loaded blanket or first wall protection is significantly below 1000 °C. Both applications offer the possibility to use oxidation resistant alloys, either because of the oxidation during normal operation, or for safety concerns in case of an accidental loss of cooling event.

The most important questions in this line of research are: What is the optimized microstructure for fusion relevant thermo-mechanical load conditions? Is it possible to increase the fracture resistance?

4.1. Production and optimization

Up to now only commercially available tungsten materials (pure W, WL10, WVM) were considered for divertor armor applications. In order to achieve an isotropic, crack-resistant material, mechanical alloying, followed by one or more HIP steps, was used to produce W–Y, W–Y_2O_3, and W–TiC alloys. It was possible to produce sub-micrometer grains in all materials. For the selection of the optimal composition specimens for various material and high heat flux tests are presently prepared.

Production of W–Si–Cr alloys by mechanical alloying was driven by the possible need for a protection material with good oxidation resistance. For this ternary alloy the self-passivation effect has been demonstrated up to about 1000 °C [18]. Up to now several production routes have been tested and the efficiency of the oxide layer has been investigated (see Fig. 2). However, further optimization work will be performed to reduce side effects like volume change and densification problems.

The thin film oxidation resistance of quaternary W–Si–Cr–Zr and W–Si–Cr–Y systems was also studied. It could be shown that these films show a better passivation behavior. The active elements (Y or Zr) improve the oxide adhesion and change the oxidation mechanism by slowing down oxidation while increasing the tungsten atomic concentration compared to the best ternary alloys.

4.2. Characterization

Besides basic material characterization in an extended high temperature range (including thermal fatigue), thermal shock tests in the operation relevant parameter range (JUDITH electron beam facility, FZJ, Jülich [19]) and thermal fatigue tests under hydrogen and/or helium beam loading (GLADIS, IPP, Garching [20,21]) with divertor relevant fluxes were carried out.

The effect of edge localized modes (ELMs) in the plasma on the commercial pure tungsten reference material (see Section 3.2) was simulated in the JUDITH facility. The incident power flux was varied between 0.15 and 1.3 GW/m^2, the test temperatures ranged from room temperature up to 600 °C, and the specimens were loaded with 100 pulses with a duration of 1 ms. Damage by surface cracks was significantly influenced by the microstructure (see Fig. 3), that is, by grain size (after annealing) and by the grain orientation. The ongoing investigations comprise an increase of pulse numbers (1000 and 10,000) as well as the study of other materials.

Pure He beam loading of tungsten specimens were carried out in the GLADIS facility. At maximum temperatures of 2100 °C (10 MW/m^2), sub-surface changes in several microns depth beyond the particle penetration depth ($\sim$100 nm) were observed (see Fig. 3). Furthermore, with actively cooled samples (maximum temperature 200 °C, 2 MW/m^2) the sputtering behavior was investigated. It clearly depends on the orientation of the grains exposing different crystallographic planes (see Fig. 3). Finally, first 10 MW/m^2 load tests with a mixed H/He (90/10) beam showed an onset of void formation at 850 °C [22].

5. Materials science and modeling

The intrinsic brittleness of tungsten is primarily due to the high activation energy of screw dislocation glide. The scatter of fracture toughness shows in addition that other factors such as grain size, texture or chemical impurities are also important. Up to now only Re is known to form a ductile tungsten alloy. Furthermore, radiation damage data – especially under divertor operation conditions

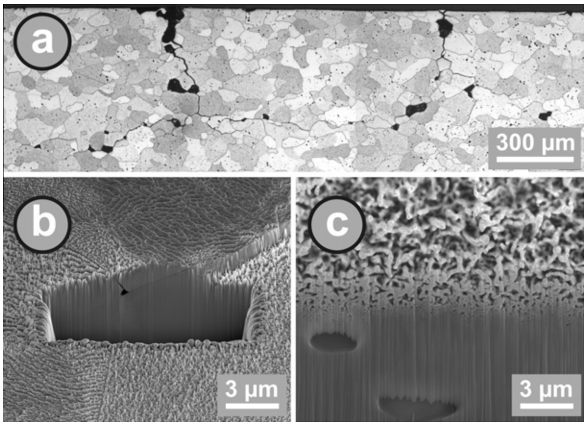

Fig. 3. High heat flux testing of tungsten armor material. (a) Surface cracks in annealed tungsten after 100 electron beam pulses (1 ms, 1.3 GW/m^2) at room temperature in the JUDITH facility. (b) Surface modification due to sputtering after helium loading (2 MW/m^2) at 200 °C (peak temperature) in the GLADIS facility. Sputtering rates as well as surface structure depend on grain orientation. (c) Sub-surface changes after helium loading at 2100 °C (peak temperature) in the GLADIS facility. Courtesy of G. Pintsuk, FZJ, Germany and H. Maier, MPI-IPP, Germany.

– is very rare. Therefore, the main objective for this research line is to assist and guide the materials development process. The basic idea is to identify the origin of the extreme brittleness of tungsten and to explore a range of potential ductilization treatments. In parallel modeling radiation effects, i.e. point defects and He/H accumulation, in bulk and sub-surface has to be performed.

The most striking questions are: What makes tungsten so brittle? Is ductilization possible besides Re addition? What is the influence of impurities and microstructure on the material behavior? How does tungsten behave under high neutron doses and after significant He/H load?

5.1. Plasticity studies

Fracture toughness tests on different tungsten grades show both intergranular and transgranular fracture behavior. Auger electron spectroscopy (AES) of fractured surfaces seems to indicate that below certain limits impurities do not influence the crack propagation resistance of grain boundaries. Surprisingly, these limits are usually fulfilled in industrial alloys. Therefore, it seems to be an intrinsic tungsten material characteristic that grain boundary fracture needs less energy than transgranular cleavage.

Further experimental work on the ductility of tungsten alloys comprises detailed analyses of the *R*-curve behavior and the studies of the dislocation structure and density effect on fracture toughness. For conditioning the microstructure (and also alloying), severe plastic deformation as well as micro sample tests are used.

5.2. Simulation and validation

In principle, a range of atomistic modeling tools can be applied to simulating tungsten alloys. That comprises (1) *ab initio* calculations of core structure and energetics of screw dislocations in W and W–Re, (2) *ab initio* and molecular dynamics energetics of He/H [23] and point defects [24], (3) molecular dynamics simulations of defect production by irradiation in the presence of He, (4) kinetic modeling of He and dpa accumulation [25], and (5) molecular dynamic simulations of the mobility of edge and screw dislocations and of their interaction with He vacancy clusters (irradiation hardening).

Various validation experiments are planned for microstructure analysis of He implanted tungsten and under dual-ion beam conditions, for studies of the recovery kinetics under thermal annealing treatments, and for *in situ* TEM observations to study dislocation dynamics and interaction with radiation defects (JANNUS facility, CEA, CNRS and University of Orsay, France).

So far first TEM studies of He bubble formation after implantation and annealing have been performed. Moreover, results for lattice relaxation and self interstitial atom (SIA) formation energies from systematic *ab initio* studies are available for all bcc transition metals [26–28] and also for W–Ta and W–V alloys. Contrary to W–Ta, in W–V relaxation is almost absent and V is expected to be more easily trapped in mixed SIAs. Therefore, the main conclusion is the prediction of a possibly better irradiation performance for W–V alloys.

6. Summary

The long-term goal of the EFDA programme on divertor materials is to provide structural and functional materials together with the necessary production and fabrication technology for helium cooled divertor components. So far the difficulties and problems connected to the development of tungsten materials for fusion applications are well known and identified. Therefore, until the end of 2012 the focus will be laid on the identification of possible limitations and show stoppers for tungsten materials and related fabrication technologies.

While fabrication issues are far advanced and well investigated, the most critical part of the programme is probably the development of a material for structural divertor parts. Joining of tungsten materials is possible, but developing low-activation brazing materials is still a problem. Testing and characterizing possible armor materials is well advanced and may lead to optimized armor materials. A complete picture of the irradiation performance of tungsten materials is not yet available. This program should provide routes in terms of chemical composition and thermal–mechanical treatments in order to improve the ductility and fracture toughness of W-alloys, and then proceed with neutron irradiation of possible candidate materials.

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