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INCORPORATION OF TUNGSTEN METAL FIBERS IN A METAL AND CERAMIC MATRIX

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Tungsten fibers have high tensile strength but a poor oxidation resistance at elevated temperatures. Using this first characteristic and to prevent oxidation of tungsten coated composite materials in which the primary requirement: reinforcement against destruction or deformation, was studied on tungsten fibers and tungsten wires which were coated by applying the metal and ceramic powders via plasma spraying device in plasma generator WSP®. Deposition took place in an atmosphere of Ar + 7 % H_2, sufficient to reduce the oxidized trace amounts of tungsten.

Key words: tungsten wires, tungsten fibers, plasma spraying, metallic coatings, ceramic coatings

INTRODUCTION

Research on materials to be used under extreme conditions, i.e. at high temperatures and pressures, has a large number of variants. While ultra - high vacuum or ultra - high pressures can certainly be considered extreme conditions, with temperature it is easier. At very low temperatures, all substances are in a solid state, where the design parameters are more readily achievable. Conversely, temperature has no upper limit and its increase leads phase changes to the liquid state, the gaseous state, and finally plasma. Today there is no problem to achieve in the plasma state, temperatures of hundreds of thousands of degrees Kelvin. The problem, however, lies with constructional materials for devices where with ultra - high temperatures must be used. The current limit of all elements in this direction is represented by tungsten with a melting point (mp) of 3 420 °C and for alloys by tantalum carbide and hafnium with melting points of approximately 3 800 °C. These data highlight the fact that above a temperature of 4 000 °C, there is nothing on Earth in a solid state that could be used to construct devices for use in extreme temperatures.

At present, the latest approach to using ultra - high temperatures is for the generation of plasma, which is necessary to begin nuclear fusion in Tokamak - type devices with a view to obtaining an infinite source of energy solutions [1, 2]. This relies on the development of tungsten – based materials, e.g. cermets which alter or improve the thermal conductivity, corrosion resistance, resistance to neutron radiation, etc. e.g. in ITER project [3 - 5]. However, these also reduce the limit for high – temperature usability. Such materials, however, can be used in many other technical sectors where it is necessary to increase mechanical strength at high temperatures and, at the same time, protect metal parts from oxidation and potential chemical corrosion.

Current suitable methods for processing materials with the highest melting points are thermal spraying, with plasma spraying having the highest high - temperature tolerance [6], or hot stamping or sintering, again with the highest limit variant being Spark Plasma Sintering (SPS) or also called Field Assisted Sintering Technique (FAST).

This work is focused on the use of the unique properties of tungsten filaments and wires which are characterized by high tensile strength up to 3,4 GPa, the highest density and smallest thermal expansion coefficient of all metals ($4,3 \div 4,6 \cdot 10^{-6}$ K^{-1}) to the reinforcement of metallic and ceramic materials with other utility properties. The incorporation of tungsten in materials with lower melting points increases their mechanical reinforcement against all kinds of stress, particularly at high temperatures. Coatings on tungsten wires and fibers also prevent oxidation, which becomes critical from 600 °C.

EXPERIMENTAL

The experiments used a tungsten substrate in the form of sheets of 1 mm thickness, a Metallwerk Plansee product, and tungsten wire of 0,8 mm diameter and tungsten filaments of a 0,02 mm diameter, both products of Osram Bruntál Ltd. Chemical composition of the substrates is shown in Table 1.

These tungsten substrates or mandrels were also used for further experiments:

a) without changing the original morphologies and chem. composition,
b) after mechanical surface treatment – abrasive blasting with corundum F 240 to R_a 7 μm – 8 μm as measured on the Mitutoyo roughness tester SJ 210.
c) after chemical treatment consisting of SiC and WSi_2 coatings. Bundles of several tens of fibers with longitudinal orientation were between electrical contacts of

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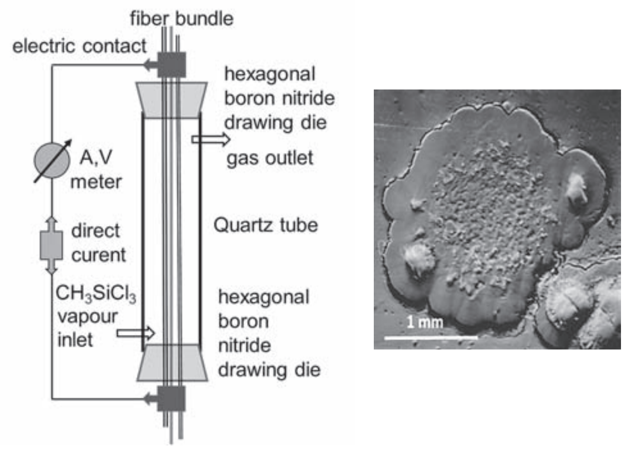
Table 1 **Chemical composition of tungsten substrates / wt. %**

	W plate	W fiber	W wire
W	99,5	99,92	96,67
Na	--	--	0,29
K	0,02	--	0,35
Si	--	--	0,36
Fe	0,31	--	0,51
Cu	--	--	0,10
Zn	--	0,07	0,04
Cr	0,03	--	--

direct current (14 V; 20 A – 40 A) incandescent in an evacuated quartz apparatus, to which were fed a methyltrichlorsilane vapour entrained with Ar carrier gas from the container, see Figure 1. Based on the deposition time, rigid rods, about 1 mm thick, covered with silicon carbide, which is used as another type of tungsten substrates, particularly to improve the adhesion of other coatings by increasing the surface roughness with a permanent chemical, rather than a mechanical, linkage, were created. A typical cross section of resulting formation is shown in Figure 1(b).

Tungsten wires of 0,8 mm diameter with a protective surface layer of WSi_2 (blended with small quantities of phase W_5Si_3), were formed by reacting the surface of a W - wire heated to about

1 400 °C in powdered silicon. The uniformity of the coating thickness was largely ensured by moving the tungsten wire between the sliding electrical contacts and hexagonal boron nitride drawing die Figure 2.



a) b)

Figure 1 (a) Scheme of SiC coatings preparation on a tungsten fibers and (b) the cross-section of the sheaf tungsten fibers in SiC matrix

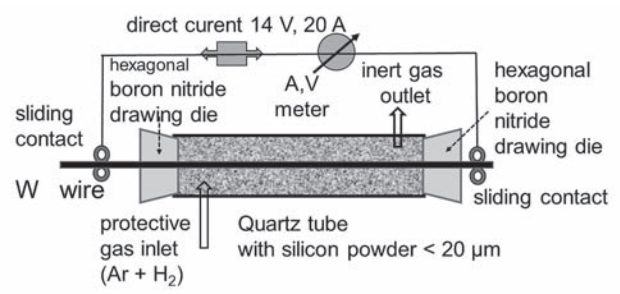


Figure 2 Scheme of WSi_2 coatings preparation on a tungsten wires

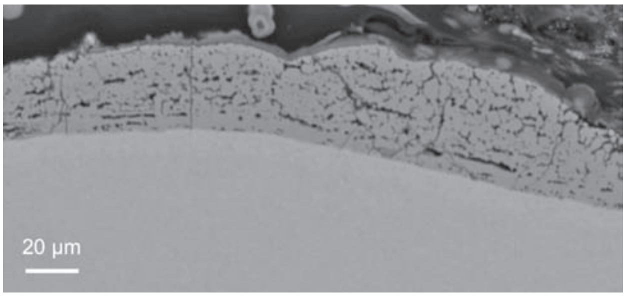


Figure 3 Cross-section of tungsten wire with WSi_2 coating

Cross - section of this type of coated tungsten substrate is shown in Figure 3.

Subsequent geometrical modification of substrates: bundles of individual tungsten fibers or wires and coated tungsten wires were assembled into parallel molds longitudinally, in the shape of a flat lattice or helically wound on an inert ceramic boron nitride or graphite substrate - mandrel.

Blank tungsten substrates were used to determine deposition conditions during plasma deposition, i. e. to determine the optimal feeding distance and scattering angle between the neutral and oxidative zones of the plasma jet. A record of the spray footprint is shown in Figure 4.

For deposition of tungsten powder with grain size classes of 40 μm - 63 μm, a WSP® - H500 plasma generator was used, working with the parameters 450 V, 320 A, 144 kW power. Successful tungsten deposition using this device is described in a number of previous works by UFP [7, 8]. The obtained materials were analyzed metallographically and SEM microscopically, adhesion of tungsten to metal materials and the ceramic matrix was measured according to EN ISO 4624.

RESULTS AND DISCUSSION

From the geometry of the spray pattern of tungsten on the flat substrate (see Figure 4) it is evident that the neutral plasma portion with molten tungsten particles has a diameter of 50 mm with a deposition distance of 300 mm, i. e., 0,038 sr. A spatial angle over 0,080 sr always leads to oxidation, which, due to the high turbulence of the plasma cannot be prevented even with very

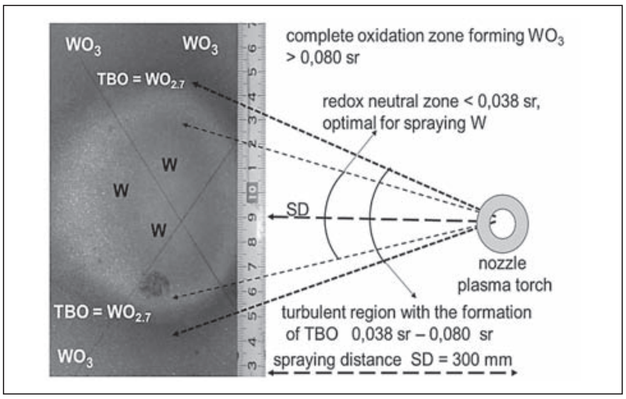


Figure 4 Evaluation scheme of footprint deposition tungsten by thermal spraying

intense shrouding [9]. That is why the deposition of tungsten on any substrate was carried out in the graphite apparatus, see Figure 5. This enables longitudinal and rotational movement of the substrate while maintaining the substrate with deposited tungsten in a slightly reducing atmosphere of Ar + 7 % H_2. Under these conditions there is no oxidation of the tungsten or its melting and the formation of splats. In this apparatus it is possible to coat tungsten fibers with other metal and ceramic materials, e. g. titanium, titanium nitride or ceramic aluminum oxides.

The first experimental variant in the preparation of the molded part was deposition of tungsten on tungsten wires with a diameter of 0,8 mm, in order to connect them and create a tube with a freely shaped profile, in this case a non-circular shape. The bundle of tungsten wires were inserted into the graphite sleeve. Figure 6 (a), (b) show the initial stage of deposition of tungsten molten splats and detail of the surface tungsten reinforcement wires.

Increasing thickness of the tungsten coating balances out waviness in the coating. Other preform variants of the tungsten substrate for subsequent plasma deposition were carried out so that, on a temporary substrate made of graphite or mechanically, readily - removable materials (e. g. hexagonal boron nitride ceramics), the tungsten wire or a tungsten filament could be once or

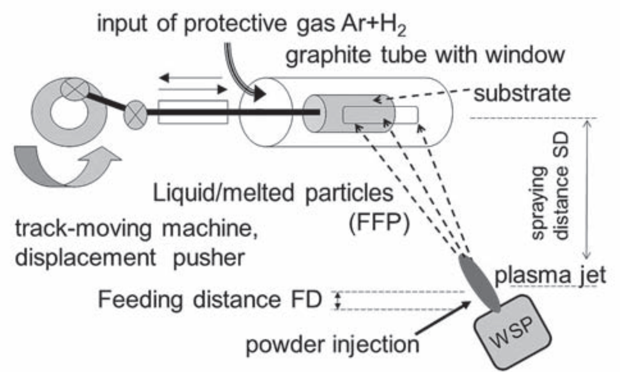
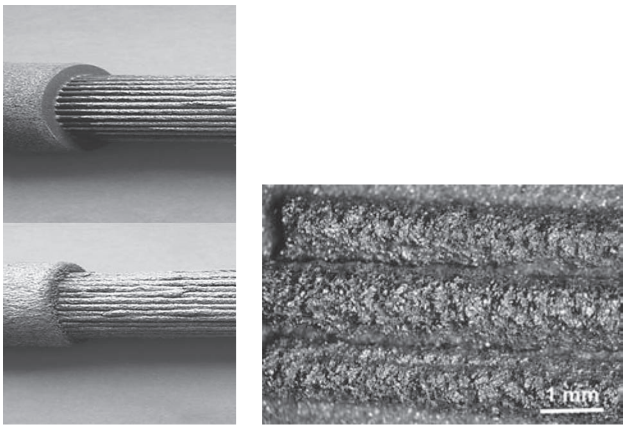


Figure 5 Scheme of plasma spraying on movable or stationary tungsten substrates in a controlled atmosphere



a) **b)**

Figure 6 (a) Deposition of tungsten on a complex armor wires and (b) the detail of the connection of individual wires

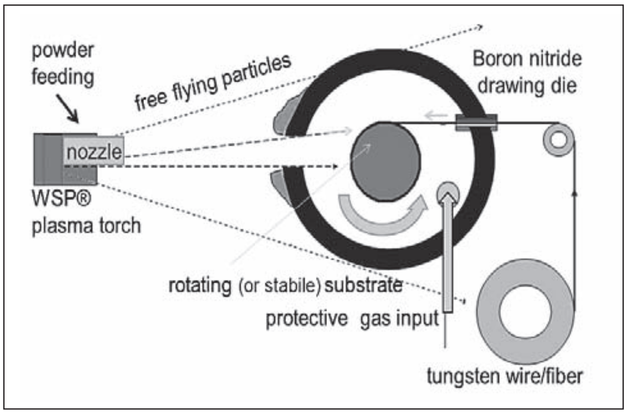


Figure 7 Scheme deposition on the moving parts of tungsten substrates

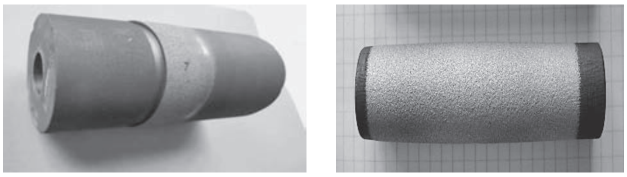


Figure 8 Examples of W-coated parts on graphite

continuously rebound with the simultaneous deposition of the feed material, see scheme on Figure 7.

Variations of continuous deposition of titanium, reactive deposition of TiN or oxide ceramics, for.

example corundum on W – fibers and wires or bars, see Figures 8 – 12, were already successfully tested using the temporary graphite mandrel [10].

CONCLUSIONS

In terms of the process of thermal spraying, it is not critical if sprayed on the flat or shaped W -substrate, parallel or lattice sorting of fibers and wires or winding it around the helix. Such modified tungsten wires fulfill a reinforcement function for the substrate under mechanical stress across the system. Because auxiliary construction materials usually do not withstand temperatures greater than 2 000 °C during atmospheric plasma spraying APS, they must be cooled or the plasma spraying process interrupted. In this case, an interlayer between which there is a strain due to temperature

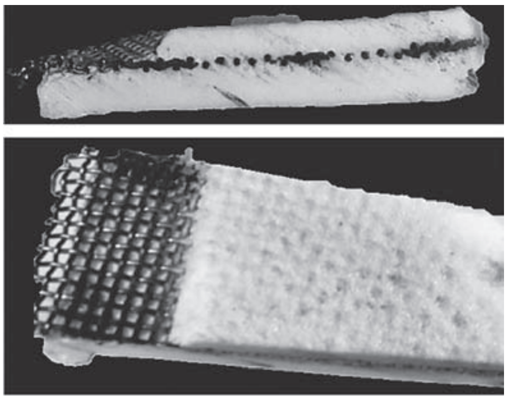


Figure 9 Details of metal bars in the corundum matrix prepared by plasma spraying

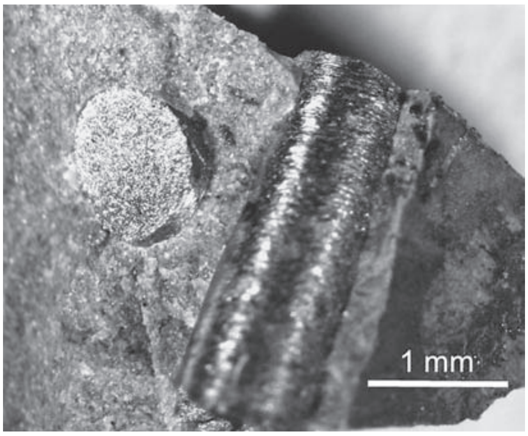


Figure 10 Detail of the building of tungsten bars into a titanium matrix

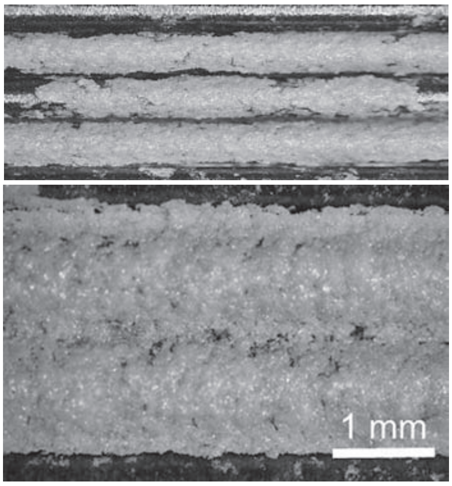


Figure 11 (a) Gradual coating of W-wires d = 0,8 mm with corundum ceramics, poor adhesion in the original, (b) improve adhesion to the WSi_2 surface

changes or to oxidation, since hydrogen of protective mixture of $Ar + H_2$ has a reducing activity from about 900 °C, is formed in the coating. The disadvantage of a smooth surface on fiber and wires is low adhesion with ceramic coatings. This problem is solved by roughening the surface via chemical reactions in the preparation stage of the fiber. In the case of a metal matrix, the adhesion value cannot be quantified, as it is always at the level of strength of the implied metal to metal diffusion bonding, thus higher (over 60 MPa) than the adhesive strength specified in EN ISO 4624 or ASTM C633 [11].

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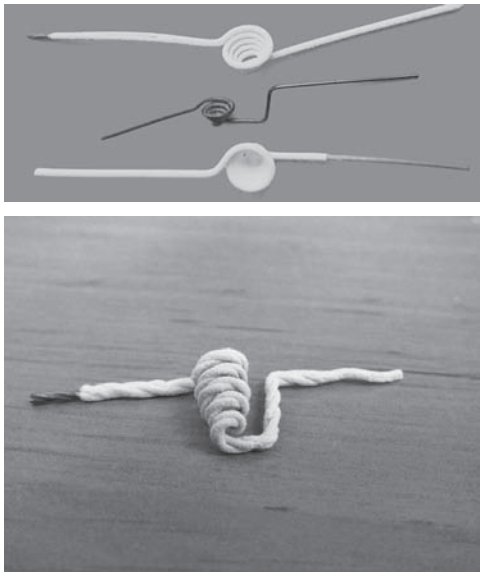

Figure 12 Example of practical application protective heating of W-coils for steam apparatus

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Note: The responsible translator for the English language is K. Štětková, CTU – Klokner Institute, Prague, Czech Republic