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Tungsten – Rhenium Alloys Wire: Overview of Thermomechanical Processing and Properties Data

Boris Bryskin
Hoskins Manufacturing Company, Inc.
Hamburg, Michigan, USA

Summary:

The scope of this study encompasses the compositional modifications of the tungsten-rhenium dual system (W-3/5Re up to W-27Re) as well as some of the tungsten-molybdenum-rhenium ternary system. The alloys of interest are considered with a specific representation of powder metallurgy route based on doped or undoped tungsten vs. vacuum melted materials. This paper constitutes an in-depth review of structural and mechanical properties and systematic compilation of challenges necessary to provide the quality consistency of severely drawn filaments. The issue of thermomechanical processing trends is addressed as an important part of W-Re fabrication technology to achieve further improvement in design properties of rod and wire.

Keywords:

Powder metallurgy, rhenium, tungsten-rhenium, wire, thermomechanical treatment, structure, mechanical properties, drawing, annealing.

1. Introduction:

Although Re is a scarce material, W-Re alloy combinations have won wide and important applications in various modern technological fields and achieved great economic benefits. Widespread applications for thermocouples, lamp filaments, electric and electronic devices, welding electrodes, etc. are in high temperature environments, where the alloys outstanding properties, such as elevated melting point and recrystallization temperature, appreciable electrical resistivity, good low temperature ductility, great creep strength, favorable wear and corrosion resistance can be put to their best usage (1-4).

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The development of W-Re wires for commercial applications persists as a source of continuing innovations in material science and manufacturing technology and consequently there is a strong engineering motivation for guiding further alloy design and fabrication improvement. Additions of 3-5% Re (all compositions in weight %) to overcome the severity of pure W embrittlement appear to be beneficial due to alloy softening, though less spectacular in their effect than the solubility-limit concentrations of 24-27% Re which significantly promote the strength and ductility increment imparted to W by solid solution strengthening.

The growing acceptance of W-3/5 Re and W-25 Re wires is primarily fueled by favorable experience as alternative thermoelements (5). The alloys of interest are generally manufactured in the USA and Europe through the powder metallurgy (P/M) route with a tolerance on the Re addition in the range of ± 0.5% or better. In order to suppress the phase formation, vacuum melting (V/M) is the most popular W-27Re production process in Russia with Re content tolerances of ± 3%. In alloys W-3/5 Re the W-base material is intentionally doped with K, Al and Si (AKS tungsten) to control recrystallization behavior. Plansee and Russians manufacture wire of such alloys in the undoped condition. There is strong incentive to achieve better material performance by addition of 10, 15, and 20% Re to W as so-called "dilute or moderate alloys",
or consideration of high Re ternary alloys with W and Mo (6). Dispersion strengthening by interstitial refractory phases (HfC, ZrC, Th O_2) sharply increases the high temperature strength of both low and high rhenium alloys and decelerates their softening upon heating (7).

Further prospects to attain property enhancements include the engineering of proper alloying, formation of optimal structural states under thermomechanical treatment (TMT), and using new manufacturing methods. An extensive survey of physical, chemical, thermal, magnetic, and selective mechanical properties was recently conducted by this author (6, 8) to provide background information on Re and its alloyed systems with Mo and W and to aid in the usefulness of the compilation as a reference source. The balance of the present paper will be concerned with the processing and resulting microstructures and mechanical properties that can be produced in W-Re wire or rod forms. This was motivated by the importance to successfully transmit current knowledge for high performance applications in leading edge technologies.

2. Compact Consolidation and Wire Fabrication:

The P/M consolidation technology of W-Re bars by solid-state diffusion involving blending of elementary powders, pressing to green compact, and high temperature sintering is already well established. Consequently there is much experience in the field. Further fabrication usually includes a combination of TMT steps such as swaging to fully densify the sintered bar and to reduce it to size more suitable for drawing, and wire drawing until the final diameter is obtained. Because of high-elevated temperature alloys strength and lack of plasticity, heating throughout swaging and drawing promotes sufficient material ductility. However, W-Re alloys are generally worked below their recrystallization temperature and development of fibrous-appearing structure is noticeable, accompanied by significant work hardening rate vs. accumulated deformation (9-13). Due to complex interaction of process details and differences in operating conditions, detailed description of manufacturing is beyond the scope of this paper.

The intermetallic compound sigma (σ) phase, with its habit of appearing in W-Re alloys near the solubility limit in stringer-like morphology, is the most likely contender for the dubious honor of causing a discouraging degree of quality variation related to longitudinal fissures which easily propagate into wire cracks (splitting). The inevitable formation of sigma phase as a row of fine scale precipitates can be mitigated by following efforts to enhance the creation of a homogeneous W-Re solid solution: mechanical alloying of a blended elemental powder mixture, coating of the W particle with a Re film, application of high temperature sintering, or proper combination of TMT parameters. However, this undesirable phase is hard to entirely eliminate by any prevention technique because the effect of repeated deformation and recrystallization cycles which cause resolution and coarsening of Re-rich strain-induced σ compound (13, 14). A practical approach in manufacturing split resistant W-25Re filaments is to produce the minimum amount of σ that can be tolerated.

An important gain in productivity as well as a significant improvement in material performance throughout manufacturing can be achieved primarily by processing enhancement. Homogenization of W-Re alloys using appropriate TMT will assure that no problems will be encountered in obtaining the desired mechanical and thermoelectric properties. Several development trends are

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known as the obvious choice, which can be described briefly below as follows.

Optimization of the input wire rod fabrication is very strong being driven by correlation between rod stock quality and fine wire processability. New, efficient rolling equipment incorporating a novel technology to replace conventional swaging/rolling operations is a great advancement. Some evidence has accumulated for the existence of favorable triaxial state of compression stress when rolling in box groove such low plasticity materials as Mo and W (15). A study conducted by the author (16) has furnished valuable data regarding use of a planetary mill to manufacture Mo rod 2.5 mm dia with upgrade drawability from as sintered bar 16 x 16 mm by one step reduction of 98%. In response to heavy rolling deformation, the structure appeared to be more uniform and a large number of dislocations created a network of fine sub-grains that impart ductility to material so it can withstand the severe distortions of wiredrawing(17).

Recent work of Russians (18) has purported to demonstrate high pay-off applications of eight-stand lengthwise rolling mill to manufacture W wire rod of 2.75 mm dia. Because the rolled wire has a stronger <110> fiber texture that leads to a higher strength and greater plasticity (19), wire manufacturing by rolling to a smaller size of about 1 mm is a challenging endeavor. A great deal of effort has been spent on the development of hydrostatic extrusion of Mo and doped W which revealed a superior room-temperature properties of rod associated with heavy polygonized uniform grain structure (15). The results of the first investigations (20) related to severe plastic deformation (SPD) indicate that equal channel angular pressing is an advanced method for processing ultra fine grained W to a grain size of about 1 mm. It is a quite attractive perspective to develop by SPD some plasticity in W-28 to 30 Re sintered parts to imbue such promising alloys with high resistance to brittleness.

“Warm” drawing of W-Re wire requires the proper combination of fabrication variables in order to avoid such detrimental affects as wire breaks, fine generation, and wear. Such variables include: reduction schedule, drawing speed and temperature, wire surface quality, back tension and lubrication. The configuration of carbide dies strongly affects friction forces and the exact amount and location of shear in the wire. In the investigation of Mo and W wire drawing of 1.0 – 0.8 mm dia at 600° - 800°C, Bryskin et al (21) reported

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that there is an optimum zone of die angles which varies from 10° to 15°, depending upon the reduction. Another study (22) locates this optimum die profile between 16° and 18°. While Perlin and Ermanok (23) stated the approach angle value of diamond die between 6° and 12° when drawing Mo 0.15 mm dia with reductions of 5 to 25%, Hallas (24) notes consistently lower drawing force with 24° die for W wire 0.25 mm dia.

3.Microstructural State and Mechanical Properties of Wrought Wire:

As a direct consequence of deformation introduced by the rotary hammering action of swaging, followed by compression forces during drawing, the structure of W-Re wires appears to consist of twisted and elongated fine fibrous grains in the longitudinal direction and heavily worked and cured grains in the cross section (13). The fibrous state of wrought doped W-3/5 Re filaments displays evidence of the string-like arrangement of the 2nd phase, which exists as small spherical potassium bubbles along the wire axis. Such microstructural refinement by severely raising strain content promotes the ductility and affects the material hardening rate even if dynamic stress relief by preheating eases residual stresses. The basic principles that govern microstructural evolution of W-Re during TMT are very similar to those related to W and data available from Briant and his collaborators (12, 25) are quite pertinent. The deformation twins are present in the alloys containing more than 23% Re (7, 13).

The tensile strength and ductility of wire are a dominant consideration in selection of drawing process parameters. Current property data of W-Re wire of different compositions is very sparse and scattered throughout the literature.

A noticeable magnitude of spread among the values is associated with individual manufacturing and testing status. The Table below lists the data, which are the mean values, based on multiple tests. The observed engineering stress-strain curves indicate that while hardening is appreciable in unalloyed W, it is essentially nonexistent in the W-Re until necking occurs and the wire maintains a reasonable ductility as indicated by elongation. The hardness is generally not uniform across wire diameter, usually being higher in the interior than near the surface. Appearance of such hardness distribution is also quite typical for drawn steel wire (23).

Table: Room Temperature Mechanical Properties of P/M W-Re Wire (26)

Alloy Diameter, mm	Wire Condition	Tensile Strength UTS ($x10^3$MPa)	Elongation (%)	Microhardness VHN
doped W-3Re	drawn 50%	2.1 – 3.0	0.5- 3.0	470 –600
0.18 –1.02	annealed	1.4 – 2.0	10 – 30	380 –460
doped W-5Re	drawn 50%	2.0 – 3.0	0.5 – 3.0	450 – 600
0.18 –1.02	annealed	1.2 – 1.8	10 – 30	350 – 430
W –25Re	drawn 40-60%	2.4 – 3.5	0.5 – 3.0	540 –750
0.05 – 1.27	annealed	1.7 – 2.3	10 – 25	470 - 530

The extent of changes in mechanical properties of W-Re throughout applications of dry wiredrawing practice is not directly proportional to the amount of drafting as it is affected by a combination of fabrication variables listed above. Figure 1 illustrates the strength of Re alloys wire as a function of drawing reduction. The strain hardening envelope (Fig. 2) addresses the issue of W-25 Re wire manufacturing with saturation in hardening when high reduction is introduced. This heavily drawn wire withstood total area reduction of 99.6% without rupture and tends to have higher strengthening increments for a given amount of cold drawing strain usually initiated from 0.254 mm (26). Such wire can be used for the component fiber in the metal or intermetallic matrix composite. Work hardening curves on Fig. 3 show the effect of wire diameter on the strength of binary and ternary alloys. W - (20-27) Re alloys surpass the alloys with 2 – 4% Re in the intensity of strain hardening (7).

4. Consideration for Wire Annealing:

For W-25 Re the primary recrystallization is complete between 1873K and 2073K and equiaxed grains are formed. With continued high temperature exposure the irregular grain growth is noticeable at 2273K and, as the grain size increases, the wire becomes less pliable at ambient temperature. Heating up to 2400 – 2600K of doped W-3/5 Re alloys produces a slight microstructural evolution while exposure at 2700K and above will remove any fibering and texturing effect. Large interlocking elongated grains (non-sag structure) develop rapidly (8, 13). Systematic analysis of the available mechanical properties data was undertaken to create a comprehensive user-expandable graphical database (Fig. 4 –18) attributed to the annealing effect.

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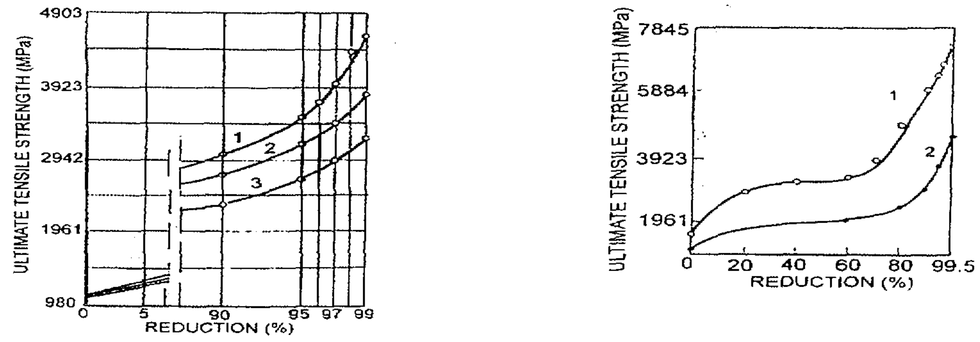

a) b)

Figure 1. Effect of work hardening through cold drawing on tensile strength of V/M Mo – 47 Re wire diameter of # 1 - 0.6 mm, # 2 – 1.0 mm and # 3 – 2.0 mm. (a) and V/M # 1 – W-27 Re and # 2 - Mo – 47 Re wire (b) (27)

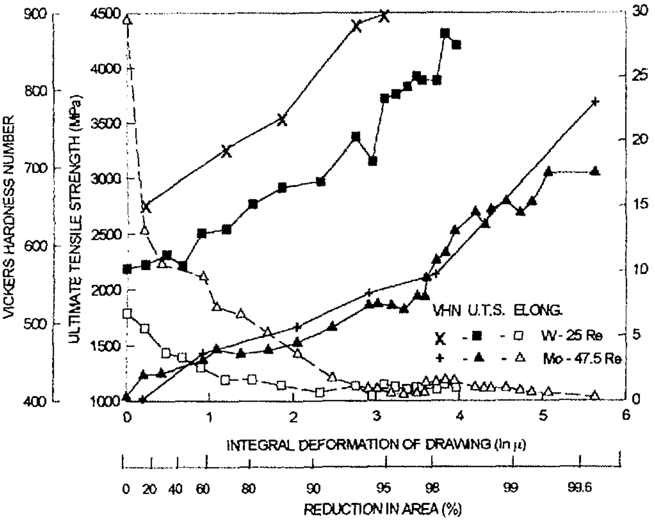

Figure 2.
Effect of deformation on the ultimate tensile strength, elongation, and microhardness for P/M wire drawn from 1.3 mm to 0.2 mm for W-25 Re and from 1.6 mm to 0.09 mm for Mo-47.5 Re. (26)

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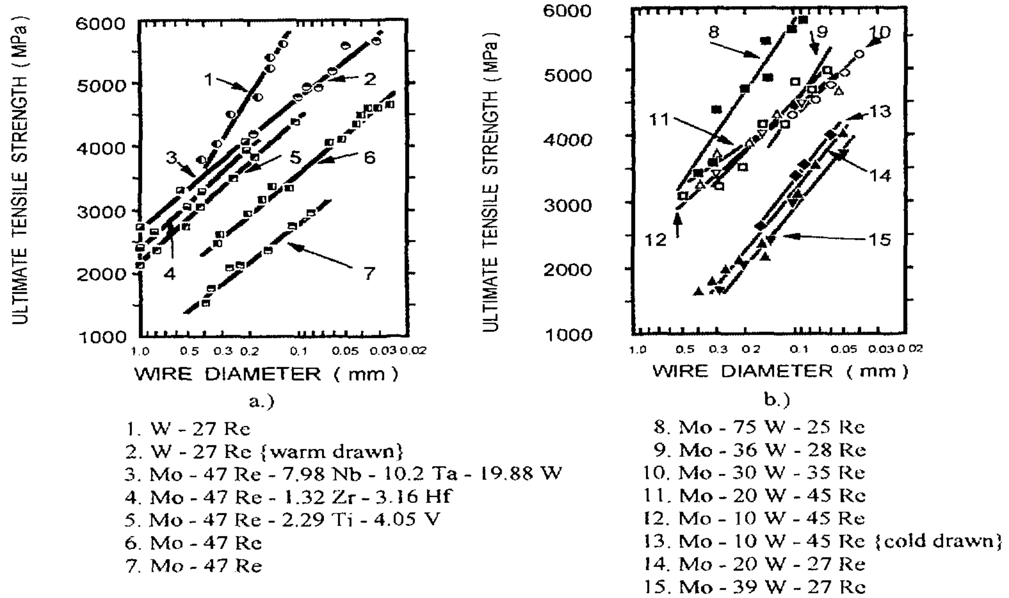
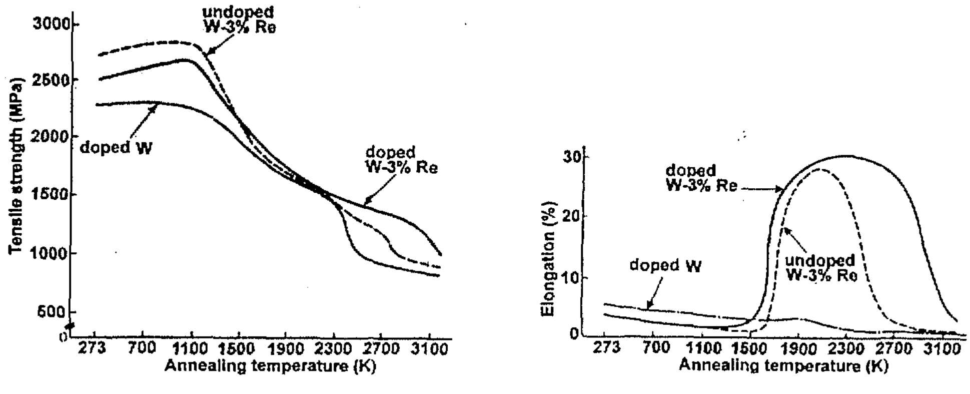


Figure 3. V/M binary with additives (a) and ternary (b) alloys of the W – Mo – Re system: Room temperature values of the ultimate tensile strength expressed as a function of the wire diameter. (28)



a) b)

Figure 4. Ultimate tensile strength (a) and elongation (b) versus annealing temperature for P/M W and W-3 Re wire diameter 0.39 mm (time of anneal 0.3 ks). (29)

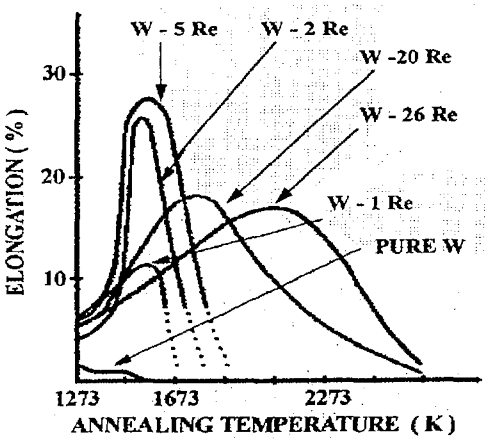
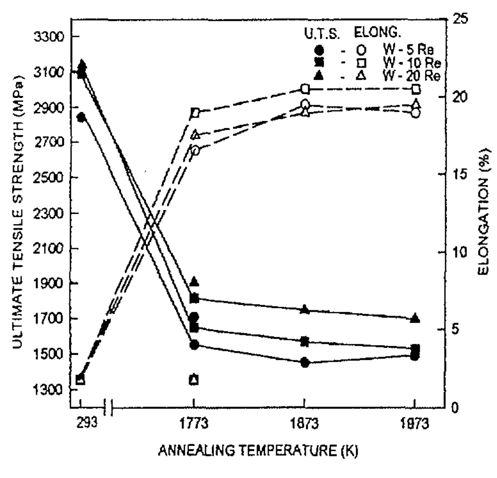


Figure 5. P/M undoped W-Re alloys – Elongation for a wire diameter of 1.0 mm as a function of the annealing temperature (time of anneal: 3.6 ks) (30)



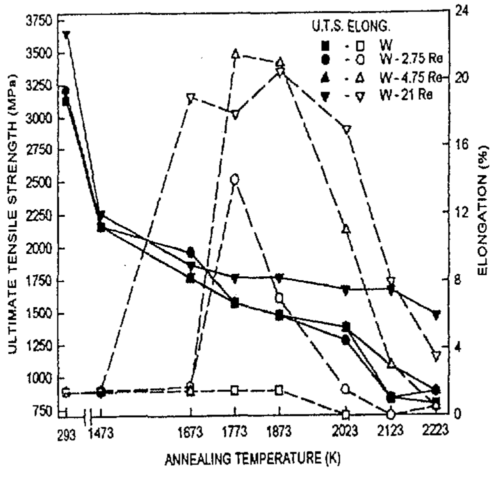


Figure 6. P/M undoped W-Re alloys -Ultimate tensile strength and elongation as a function of the annealing temperature and time of exposure of wire with a diameter of 0.34 mm. (time of anneal: 0.9 ks; at 1773 K for unlined symbols time of anneal: 15 s) (31)

Figure 7. P/M undoped W-Re alloys - Ultimate tensile strength and elongation for a wire diameter of 0.1 mm as functions of the annealing temperature. (time of anneal: 1.2 ks) (9, 32)

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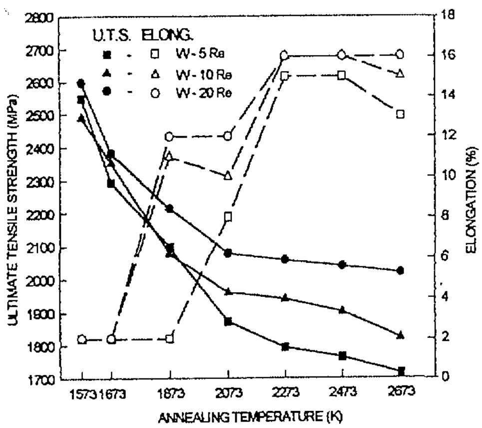

Figure 8. P/M undoped W-Re alloys (thoriated – 2.0% ThO_2) – Ultimate tensile strength and elongation for a wire diameter of 0.15 mm as a function of the annealing temperature. (time of anneal: 0.9 ks) (10)

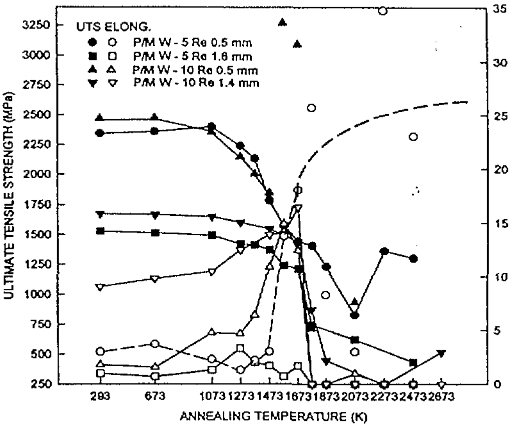

Figure 9. P/M W-Re alloys – Ultimate tensile strength and elongation for a wire diameter of 0.5 mm, 1.4 mm, and 1.6 mm as a function of annealing temperature. (time of anneal: 3.6 ks) (33)

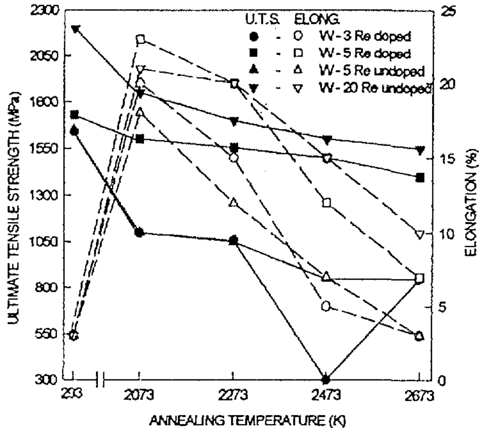

Figure 10. P/M doped and undoped W-Re alloys - Ultimate tensile strength and elongation for wire diameter of 0.35 mm as a function of the annealing temperature (time of anneal: 0.9 ks) (34)

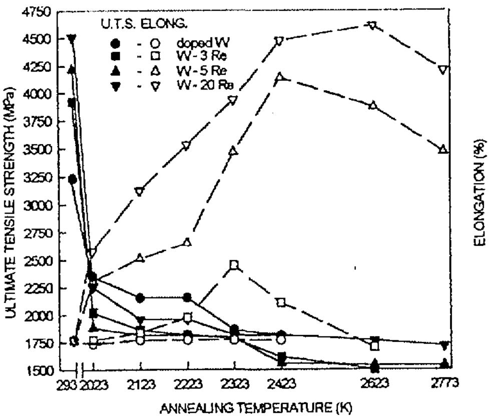

Figure 11. P/M doped W-Re alloys- Ultimate strength and elongation for wire as a function of the annealing temperature. (time of anneal: 0.9 ks) (10)

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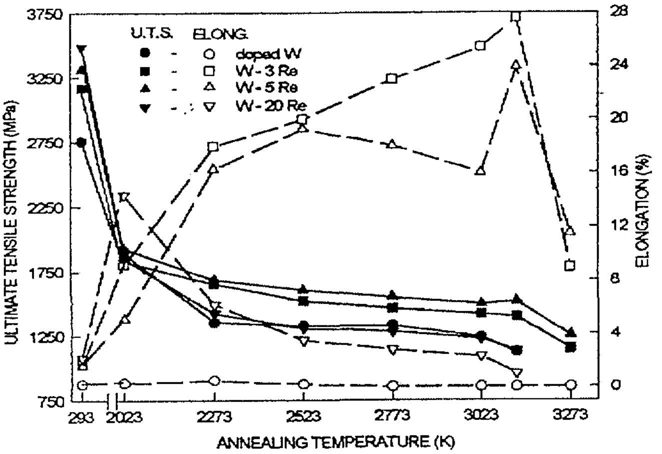
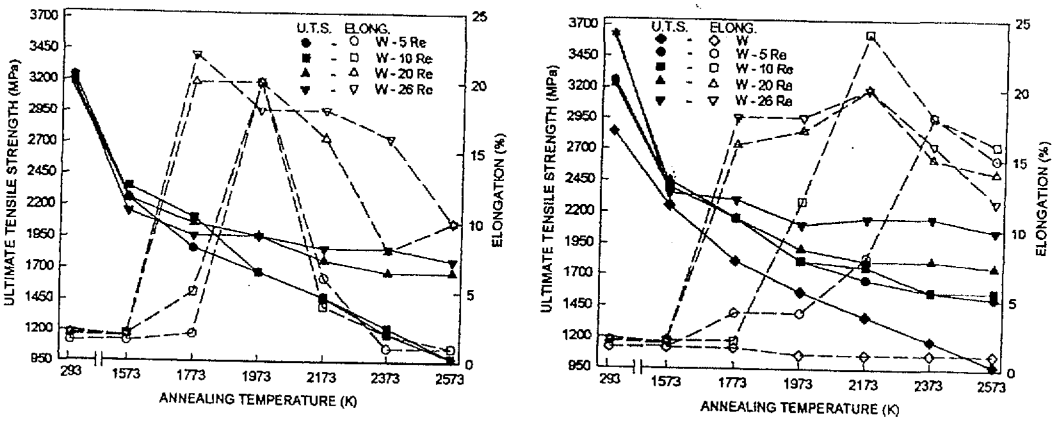


Figure 12. P/M doped W-Re alloys- Ultimate tensile strength and elongation for a wire diameter of 0.2 mm as a function of annealing temperature. (time of anneal: 0.18 ks) (35)



a) b)

Figure 13. P/M undoped (a) and doped (b) W-Re alloys – Ultimate tensile strength and elongation for wire diameter of 0.1 mm as a function of the annealing temperature. (time of anneal: 0.9 ks). (36)

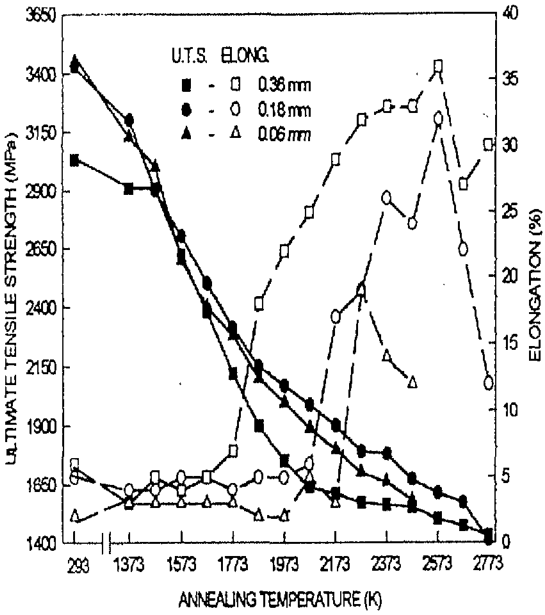


Figure 14. Ultimate tensile strength and elongation for P/M doped W-3 Re wire as a function of the annealing temperature. (time of anneal: 30 s) (37)

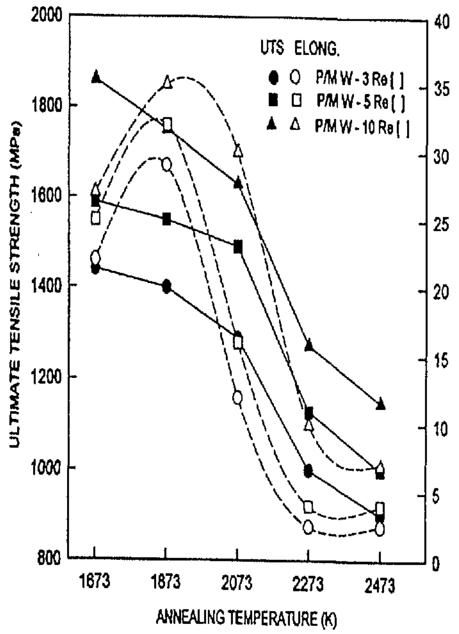


Figure 15. P/M doped W-Re alloys – Ultimate tensile strength and elongation for wire diameter 0.5 mm as a function of annealing temperature (time of anneal: 0.6 ks) (38)

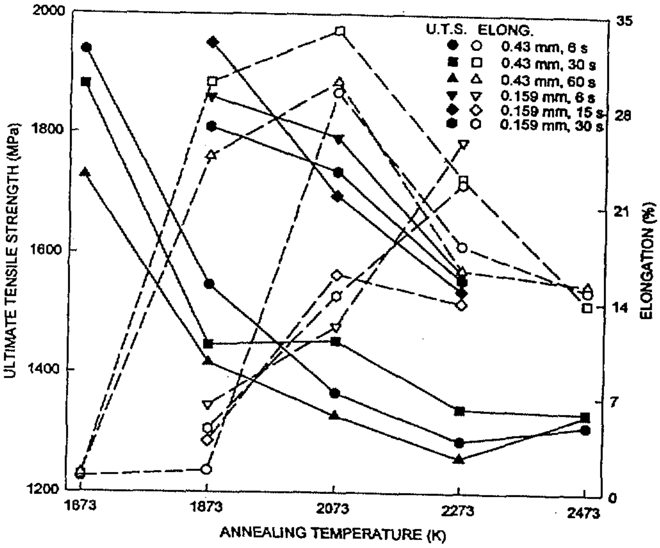


Figure 16. Ultimate tensile strength and elongation for P/M doped W-3 Re wire diameters of 0.43 mm and 0.159 mm as a function of the annealing time and temperature. As worked wires present strength 2240 MPa with elongation 1.68% for diameter 0.43 mm and 2081 MPa, 2.08% for diameter 0.159 mm (26)

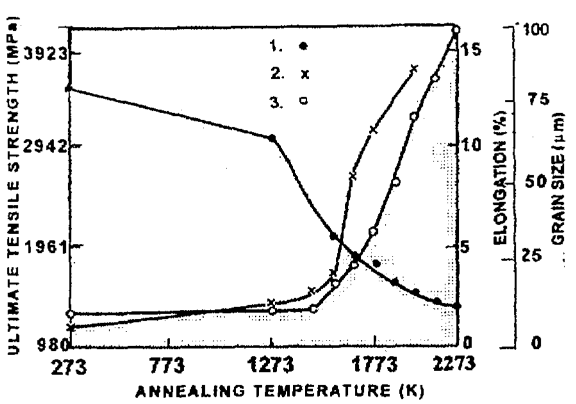
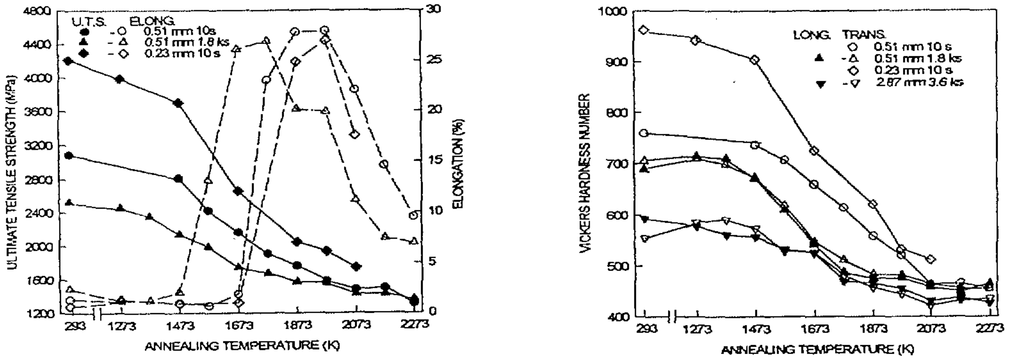


Figure 17. Ultimate tensile strength (1), elongation (2) and grain size (3) versus annealing temperature for V/M W – 27 Re wire diameter of 0.24 mm with reduction degree 94% (time of anneal in vacuum: 1.8 ks) (39)



a) b)

Figure 18. Ultimate tensile strength, elongation (a) and microhardness (b) for P/M W – 25 Re wire diameters of 2.87 mm, 0.51mm, and 0.23 mm as a function of the annealing temperature. (time of anneals: 10 s, 1.8 ks and 3.6 ks (26)

Annealing operations for W-Re wire are highly proprietary and application specific. With the exception of the annealing treatments during swaging, all in-process annealing of wires is employed essentially to remove strain-induced residual stresses and restore the optimum ductility exhausted by heavy reduction. The stress-relief operation is more amenable to maintain the drawn texture by avoiding recrystallization because of difficulties of handling recrystallization embrittled wire and resulting of wire tears and breaks.

A substantially uniform residual fine-fibred structure, slightly modified by decay, with evidence of partially recrystallized nuclei is typical for annealed wire in the Table. Continuous annealing in the temperature range of 1800 - 2400K is a practical heat-treatment. Such an intermediate processing step is required more often for the doped material, which is more susceptible to loss of low-temperature ductility of thin wire. The dissolution of subsurface contamination before annealing appears to be quite beneficial to the subsequent wire ductility. High purity dry hydrogen is the preferred furnace atmosphere because it improves surface cleanliness by reducing any oxide film.

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