Concerning Sigma-Phase in Molybdenum-Rhenium Alloys

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Molybdenum-rhenium alloys containing 40 to 48% Re by weight are commonly used for many high-technology applications. Higher rhenium contents bring the alloys into a two-phase region with the presence of brittle σ-phase, an intermetallic compound with the approximate composition of Mo_2Re_3. Alloys with lower rhenium contents do not possess sufficient low-temperature ductility and high-temperature strength. Available phase diagrams for the molybdenum-rhenium system contain conflicting information about the single-phase/two-phase borderlines. Further, the formation of σ-phase and its influence on mechanical properties depends on forming process and heat treatment, diffusion rate, precipitation mechanism, and morphology. Very few data on these factors are available in the literature. Three alloys were selected for study of the formation of σ-phase: Mo-41Re, Mo-44.5Re, and Mo-47.5Re. Applying different production parameters, especially heat treatments, the properties of the alloys were evaluated using microstructure examinations, electron probe microanalysis, and hardness testing. It was found that any harmful influence of σ-phase can be largely eliminated by properly adjusting and controlling the processing parameters. Modification of the chemical composition allows the presence of σ-phase to be avoided with no deterioration of low-temperature properties.

1. Introduction

THE so-called rhenium effect has long been recognized and studies by many researchers as one possible method for improving the strength, plasticity, and weldability of molybdenum, for lowering its ductile-to-brittle transition temperature (DBTT) in the wrought condition, and for reducing recrystallization embrittlement. Recent work (Ref 1-3) has described methods of fabrication and some of the physical and mechanical properties of the most common molybdenum-rhenium alloys containing 5, 41, and 47.5% Re (all in weight percent), which have been available since about 1960. Although some interest has been devoted to the phenomenon of alloy softening at low rhenium concentrations, the major benefits of rhenium additions are to be found at the highest possible concentration.

Savitskii and coworkers (Ref 4) were the first to construct a molybdenum-rhenium phase diagram (Fig. 1). The system has a large region for solid solution of rhenium in molybdenum, extending to 58% Re at the peritectic point and narrowing to 46% Re at 1373 K. Other investigations (Ref 4) show similar data for the solubility limit for rhenium in molybdenum: from 59% at 2713 K to 44% at 1173 K (Knapton) and from 59% at 2778 K to 46% at 1473 K (Dickinson and Richardson). Because the rhenium solubility drops with decreasing temperature, molybdenum-rhenium alloys containing about 46% or more rhenium may be subject to precipitation of the hard and brittle σ-phase, which has an approximate composition of Mo_2Re_3 and a tetragonal lattice.

The existence of σ-phase in molybdenum-rhenium alloys has been indicated in several papers. Jaffee et al. (Ref 5) stated that a small amount of σ-phase is present at 51% Re and in higher amounts at 56% Re. Maykuth et al. (Ref 6) concluded that alloys containing 45% Re or more are subject to σ-phase precipitation. Shields (Ref 7) noted that fine second-phase particles are present in Mo-47.5Re even in the as-rolled condition and that the maximum temperature for the two-phase structure is approximately 1623 K. It is apparent in the study by Falbriard et al. (Ref 8) that σ-phase was detected in very small quantities in Mo-41Re as well. These contradictory reports in relation to the maximum rhenium content without σ-phase initiation can
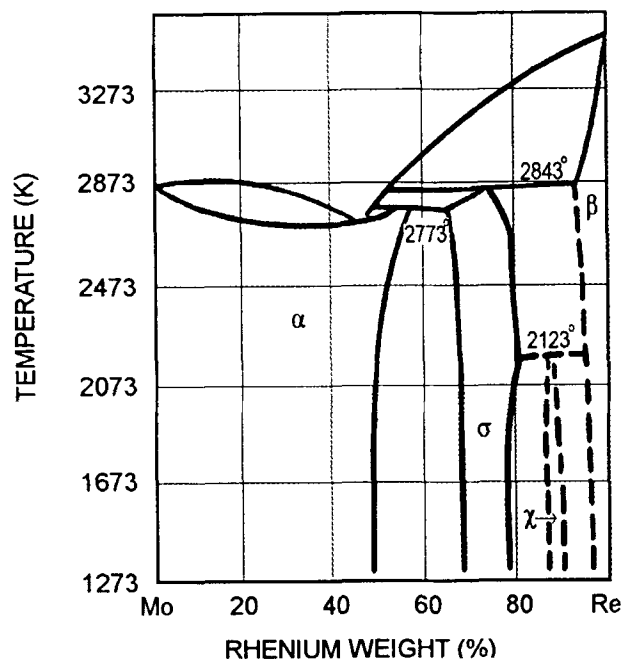

Fig. 1 Phase diagram for the molybdenum-rhenium system. Source: Ref 4

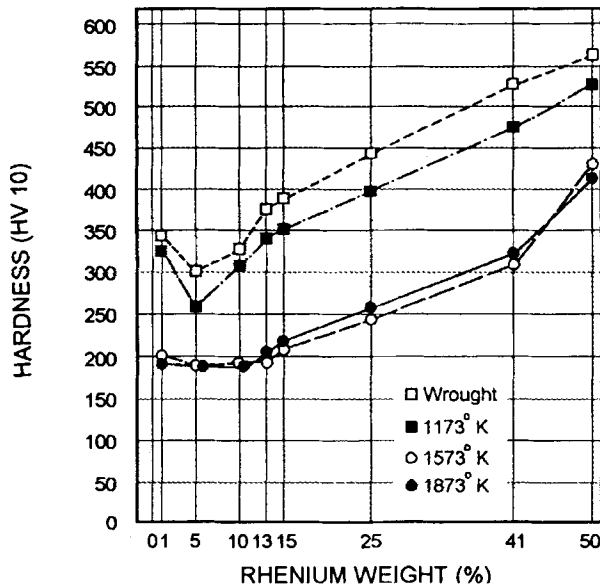**J.-C. Carlen** and **B.D. Bryskin,** Sandvik Rhenium Alloys, Inc., Elyria, OH 44036, USA

Fig. 2 Hardness as a function of rhenium content in molybdenum-rhenium alloys. Hot-rolled sheet, 1.4 mm thick, as wrought and after annealing at different temperatures in hydrogen for 3.6 ks. Source: Ref 13

be explained by conflicting information about the single-phase/two-phase borderlines in the phase diagram and differences in production parameters, particularly heat treatments.

The morphology of the second-phase precipitates is that of well-rounded particles distributed randomly in the matrix, although a common feature is a tendency for the precipitates to form stringers oriented in the working direction of the material (Ref 7, 9). Kish (Ref 10) showed the shape of the σ-phase in Mo-48Re as platelike, but a close study (Ref 11) has proved these lamellar lines in the structure to be twin lines. Finally, no evidence has been reported indicating a continuous film or high density of second-phase particles at grain boundaries or a tendency toward intergranular brittleness in Mo-47.5Re.

The presence of σ-phase in molybdenum-rhenium alloys might be expected to increase strength and decrease formability. However, at room temperature cast materials with 44 to 51% Re have almost the same hardness of 350 to 400 HV (Ref 5, 12); other authors (Ref 4, 6, 7) have demonstrated the possibility of increasing the hardness up to 550 HV by cold working. According to Fig. 2 (Ref 13), there is a small variation in the hardness of powder metallurgy alloys with between 41 and 50% Re in the wrought condition, but a very large difference after recrystallization. The cause for such a hardening effect is difficult to explain. Other authors (Ref 6, 7, 11) have shown the hardness values of molybdenum alloys containing 45 to 48% Re and annealed at 1673 K and above to be about 280 to 320 HV, which corresponds to those given for Mo-41Re in Fig. 2. In alloys with very high rhenium contents (50 to 51%), the hardness after heat treating is higher—for example, 330 to 360 HV for 0.1 mm foil after annealing at 1973 to 2373 K (Ref 4) and 435 HV for 1.5 mm diam wire annealed at 1873 K (Ref 6). There is no indication in the literature that the presence of σ-phase in Mo-47.5Re alloys causes precipitation hardening.

The possible presence of σ-phase does not adversely affect the ductility of the Mo-47.5Re alloy, because its DBTT is depressed to a very low temperature (Ref 3, 7). In the initial study of the ductilizing influence of rhenium, Geach and Hughes (Ref 12) and later Jaffee et al. (Ref 5) concluded that the Mo-50Re alloy was so ductile that it could be rolled at room temperature to a reduction of about 90% without intermediate annealing. Ample evidence for this was furnished by the successful fabrication of products by rolling, drawing, or swaging. Bryhan (Ref 14) confirms the remarkable improvement in ductility for TZM welds using Mo-50Re filler material. Powder metallurgy Mo-47.5Re has very good weldability, and sound, defect-free electron-beam welds in Mo-47.5Re sheet have been reported (Ref 11). Another joining technique utilized in aerospace applications is solid-state diffusion bonding, which makes it possible to prevent extensive recrystallization and/or grain growth (Ref 15). In addition to its attractive elevated-temperature mechanical properties and good ductility at cryogenic temperatures, its amenability to diffusion bonding is an important reason for the high interest in the Mo-47.5Re alloy from the National Aerospace Plane program (Ref 7, 16).

The kinetics of σ-phase formed by solid-state diffusion in molybdenum-rhenium alloys has not been fully investigated. According to existing theories (Ref 17, 18), the rate of homogeneous nucleation in these modestly supersaturated alloys is very small. Temperature-initiated heterogeneous decomposition causes secondary precipitates to nucleate and grow by diffusion, predominantly by pipe diffusion along wide channels of dislocations. A grain boundary is a preferred site for precipitation compared to the inside of a grain. Twin lines developed in Mo-47.5Re alloys are also favored locations for nucleation of σ-phase. This theory is supported by evidence (Ref 6) that σ-phase in Mo-50Re will precipitate during high-temperature tensile testing and slightly increase the tensile strength at testing temperatures of 1073 to 1273 K.

It is known that stress-relieving and annealing treatments at temperatures of 1373 to 1673 K accelerate the formation of σ-phase (or additional amounts of σ-phase) in work-hardened molybdenum alloys containing 47 to 50% Re (Ref 19-21). In addition to some older information (Ref 4, 6, 13) on the relation between hardness and annealing temperature, Shields (Ref 7) has recently presented data showing a sharp reduction in hardness due to stress relieving when annealing cold-worked Mo-47.5Re at temperatures of 1173 to 1573 K. At higher temperatures (1573 to 1873 K), primary recrystallization will occur (Fig. 3). A study by Morito (Ref 22) of molybdenum alloys containing 15 to 30% Re shows exactly the same stress-relieving and recrystallization pattern as for the Mo-47.5Re alloy (Fig. 4). The alloys studied by Morito are σ-phase free, so it can be stated with certainty that σ-phase precipitation has a very minute influence, if any, on the softening process for Mo-47.5Re alloys.

Although σ-phase will slowly dissolve at temperatures above 1573 K, traces of σ-phase remain in the structure after annealing at 1873 K for up to 10 ks. The most reasonable explanation for this is that in the absence of a dislocation substructure after recrystallization only slow bulk diffusion will effectively homogenize the alloy (Ref 7, 17).
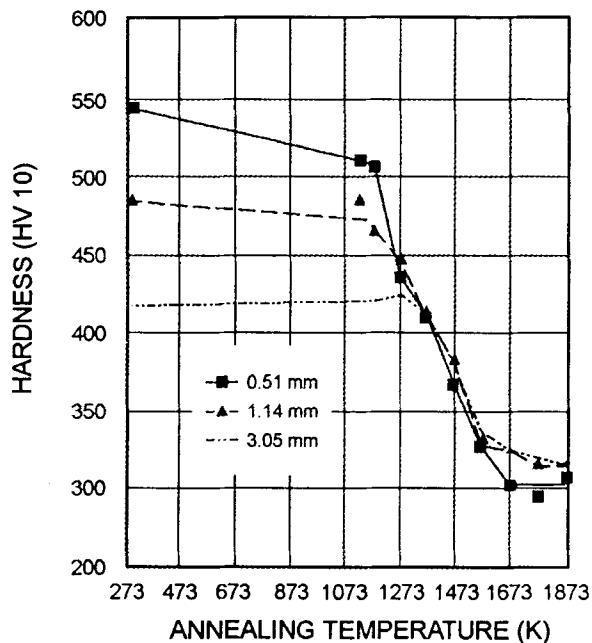
Fig. 3 Hardness of Mo-47.5Re alloy as a function of gage thickness and temperature after annealing in hydrogen for 3.6 ks. Source: Ref 20
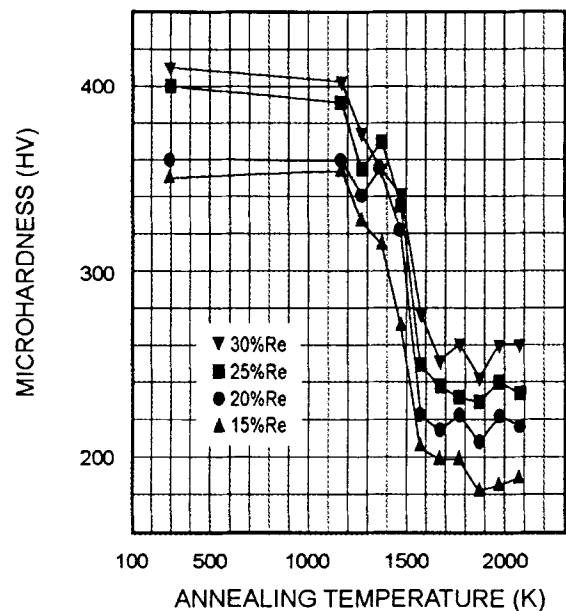

Fig. 4 Microhardness of molybdenum-rhenium alloy sheet, 1.0 mm thick, as a function of temperature after annealing in vacuum for 3.6 ks. Source: Ref 22

It is important to note that most of the above data are reported for conventionally prepared alloys. Diffusion-sintered alloys are then produced from a mixture of elemental powders; therefore, large rhenium local-concentration gradients can initially exist in the material (Ref 23). The advantage in using prealloyed powders is the reduction of σ-phase content when making Mo-47.5Re shapes for critical applications. The homogeneity of the alloying elements is achieved by coating the molybdenum grains with a rhenium film. Finer grain sizes and increased as-sintered density at lower sintering temperatures can also be achieved using coated powder (Ref 24).

Carlen (Ref 25) has suggested the development of an alloy having a rhenium content of 44.0 to 45.0% to decrease the risk of σ precipitation without affecting the high-temperature strength and low-temperature ductility of the alloy. Further investigations are required to verify the properties of this alloy.

The current study is a result of increasing interest in molybdenum alloys with high rhenium contents. A review of the literature has prompted a thorough study in order to:

- Detail the changes that occur in the σ-phase content when the rhenium concentration varies from 41 to 47.5% using conventional and coated powder materials
- Provide information on the morphology and other features of σ-phase formation and distribution during deformation/annealing procedures
- Determine the kinetics for σ-phase precipitation and dissolution during long-time, thermally activated processes

2. Materials and Experimental Procedures

Conventional molybdenum alloys with rhenium contents of 41, 44.5, and 47.5% were made using molybdenum powder of

99.9% purity, –250 mesh (Osram Sylvania) and rhenium powder of 99.99% purity, –325 mesh (Sandvik Rhenium Alloys). The Mo-47.5Re alloy was also made using the same molybdenum powder, which was then coated with a film of rhenium according to a proprietary process.

In the primary study, sheet bars were pressed, presintered, and sintered. They were warm rolled to a thickness of 1.75 mm and annealed at 1873 K, cold rolled to 1 mm and annealed at 1873 K, then cold rolled to 0.5 mm and annealed at 1693 K. The Mo-41Re alloy was warm rolled at 750 K directly from 1.75 to 0.5 mm without intermediate annealing and then heat treated at 1693 K. All annealing was performed in a hydrogen atmosphere with a dew point of less than 150 K.

To study the formation of σ-phase in Mo-47.5Re, two series of experiments were performed. Sheets with a thickness of 0.5 mm were heat treated at 1873 and 2073 K for 1.8 ks and then reheated at 1273 to 1673 K for 25 ks. After microscopic investigation, samples reheated at 1473 K for 25 ks were selected to check the microstructure for σ-phase using an electron probe microanalyzer (EPMA) equipped with energy-dispersive and wavelength-dispersive x-ray spectrometers. Inverted specimen current and secondary electron detectors were used for imaging to show sample morphology and the location of σ-phase particles. This study was made by Micron Inc. (Ref 26). Further, 1.9 mm thick sheets heat treated at 2073 K for 1.8 ks were cold rolled to 0.85 mm and then heat treated at 1273 to 1873 K for 3.6 ks.

Finally, to verify the softening and recrystallizing of cold-worked Mo-47.5Re alloys, cold-rolled sheets with a thickness of 0.25 mm (50% reduction) and tube samples with an outer diameter of 6.4 mm and a wall thickness of 0.5 mm (40% reduction) were annealed for 1 ks at temperatures between 1273 and 1973 K and compared with the 0.85 mm thick sheet.

Table 1   Microstructure/hardness characterization of molybdenum alloys containing 41, 44.5, and 47.5% Re

Condition and characteristics	Mo-41Re	Mo-44.5Re	Mo-47.5Re	Mo-47.5Re coated powder
As-sintered bar				
Density, %	96.27	96.13	96.73	95.64
Hardness, HRA	58.3	58.8	59.4	59.5
Hardness, HV(a)	244/264	261/260	255/245	248/253
ASTM grain size No.	6-2	6-2	6-3	6-2.5
σ-phase	None	None	None	None
As-warm-rolled plate, 70% reduction				
Hardness, HRA	70.2	71	72	71.5
Hardness, HRA(b)	61.5	62.3	62.5	62.9
Hardness, HV(a)(b)	257/265	282/280	287/294	283/280
ASTM grain size No.(b)	7-5	7-5	7-6	8-6
σ-phase	None	None	Yes	Yes
As-cold-rolled plate, 43% reduction				
Hardness, HRA	...	70.8	71.6	71.6
Hardness, HRA(b)	...	62.5	62.5	62.5
Hardness, HV(a)	...	394/406	402/424	...
Hardness, HV(a)(b)	...	284/279	301/294	325/334
ASTM grain size No.(b)	...	7-5	7-5	P.R.
σ-phase	...	None	Yes	...
σ-phase(b)		None	Yes	Yes
As-rolled sheet(c), 50% reduction				
Hardness, HRA	71	71.6	72.2	72.3
Hardness, HRA(b)	64	64.8	65.5	66.5
Hardness, HV(a)	434/397	414/404	403/413	416/433
Hardness, HV(a)(b)	315/301	308/319	317/310	331/312
ASTM grain size No.(b)	8-6	8-6	8-6	9-8
σ-phase	None	None	Yes	Yes

(a) Numerator, longitudinal section; denominator, transverse section. (b) Data in as-annealed condition. (c) Warm rolling for Mo-41Re; cold rolling for other alloys

Density measurements were performed according to ASTM B 328. Metallographic samples were made according to ASTM E 3-80 and prepared using procedures described in Ref 9. Grain size determinations were made according to ASTM E 112-88. Vickers microhardness testing was performed according to ASTM E 92-82 (load, 0.5 kgf; dwell time, 15 s) in transverse and longitudinal sections. Rockwell hardness testing was performed according to ASTM E 18-93 using five indentations on the sheet surface per sample.

3. Results and Discussion

3.1   Microstructures and σ-Phase Formation

Table 1 summarizes the data obtained during each step in the fabrication of the investigated alloys. Density measurements confirmed that as-sintered materials had density values falling in the range of 96.2 ± 0.5% of the theoretical. All pores appeared to be randomly distributed with rounded pore edges. The structure showed equiaxed, uniform grains, with a size of 45 to 180 µm (Fig. 5). Good homogeneity of the powder mixtures combined with high-temperature solid-state diffusion sintering resulted in good densification and a structure free of σ-phase. The microhardness of the sintered alloys was found to be in the 245 to 265 HV range, while Rockwell hardness meas-
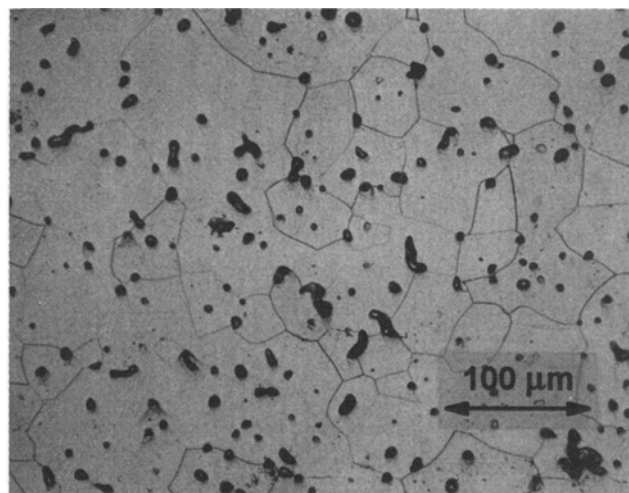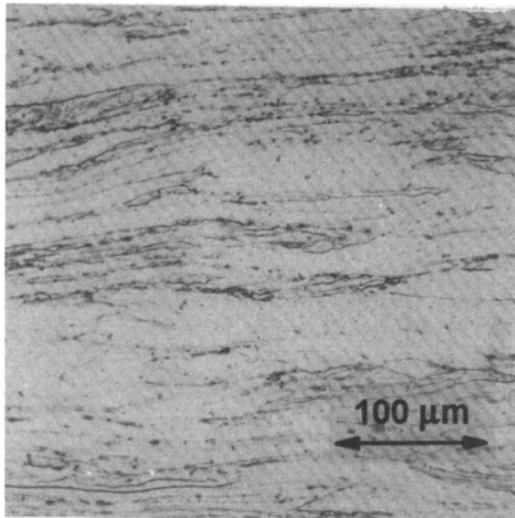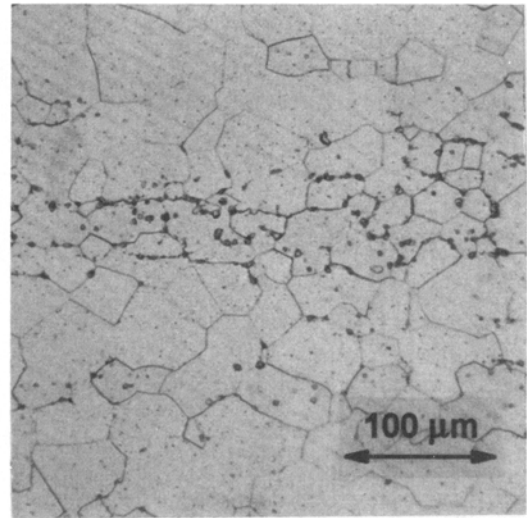

Fig. 5   Microstructure of as-sintered Mo-47.5Re flat bar (96.73% dense). 200×

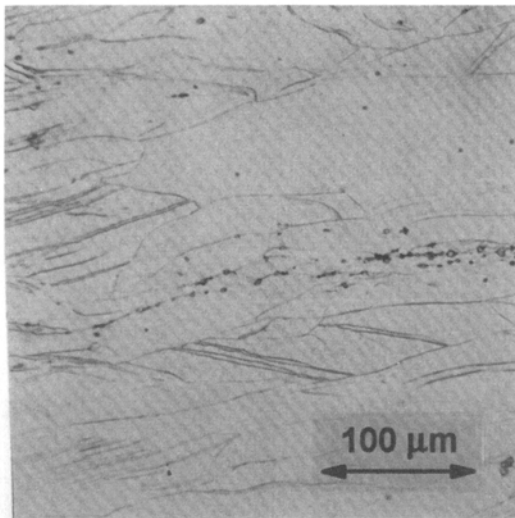urements showed a trend to slightly higher HRA values for the alloys with rhenium contents of 47.5%.

The microstructures and thus the properties of the alloys were strongly affected by the processing methods. The density was improved to a highest value of 99.98% of the theoretical
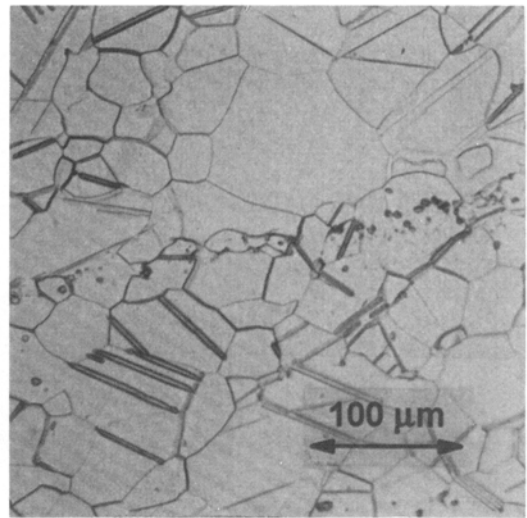
(a)

(b)

(c)

(d)

Fig. 6 Microstructure of the Mo-47.5Re blended powder alloy. (a) As warm rolled to 1.75 mm. (b) As warm rolled and annealed. (c) As cold rolled to 0.5 mm. (d) As cold rolled and annealed. All at 200×

during high-temperature rolling with a 70% overall reduction. Manufacturing by warm and cold rolling elongated the grains and aligned the grain boundaries to form a strain-hardened fibrous structure (Fig. 6a and c). Twins were formed in many grains in the alloys containing 44.5 or 47.5% Re. Both microhardness and Rockwell A values increased significantly with the degree of deformation and maintained about the same value after each processing cycle (Table 1). The structure and hardness after high-temperature rolling with preheating to 1673 to 1723 K showed that the materials had been warm rolled rather than hot rolled, which has also been observed for other refractory metals (Ref 27). Recrystallization and grain growth, up to 22 to 65 μm, occurred rapidly during annealing (Fig. 6b and d), with an appreciable drop in hardness. The results show that increasing the rhenium content from 41 to 47.5% does not have a pronounced effect on room-temperature hardness either before

or after annealing. The microhardness data do not disclose any noticeable longitudinal transverse anisotropy in rolled molybdenum-rhenium sheet, which would be the case for pure rhenium (Ref 28).

Metallographic examination of the Mo-41Re and Mo-44.5Re specimens revealed traces of a second phase. However, these traces were too scant for proper identification using optical microscopy. On the basis of the data presented, it appears that 44.5% Re is the solubility limit in the regular molybdenum-rhenium system at about 1473 K to room temperature. The microstructure displays evidence of σ-phase in blended Mo-47.5Re after warm rolling (Fig. 6a). These fine particles are uniformly distributed throughout the deformed matrix and are difficult to distinguish. Material in the annealed condition (Fig. 6b) shows an increase in the amount of σ-phase. These particles precipitated in connection with annealing occasion-
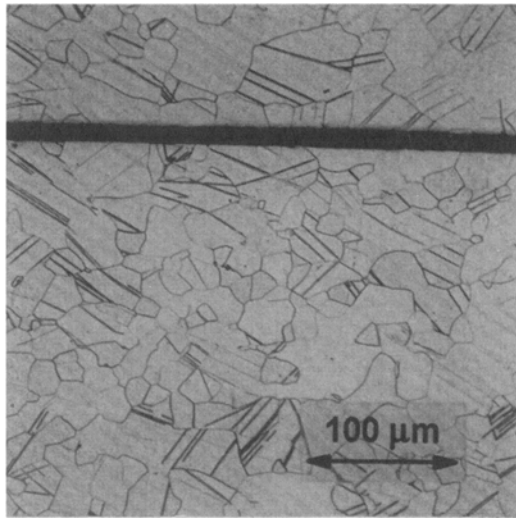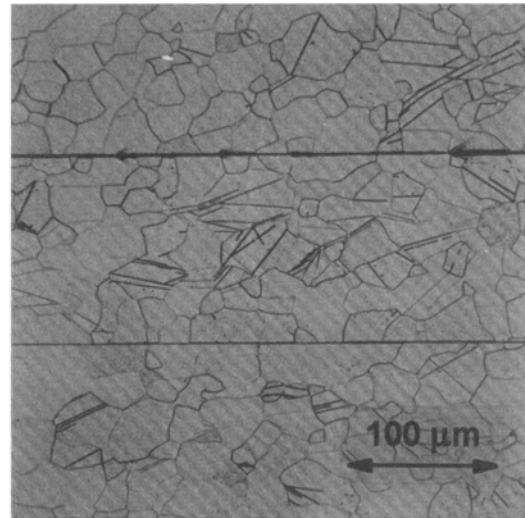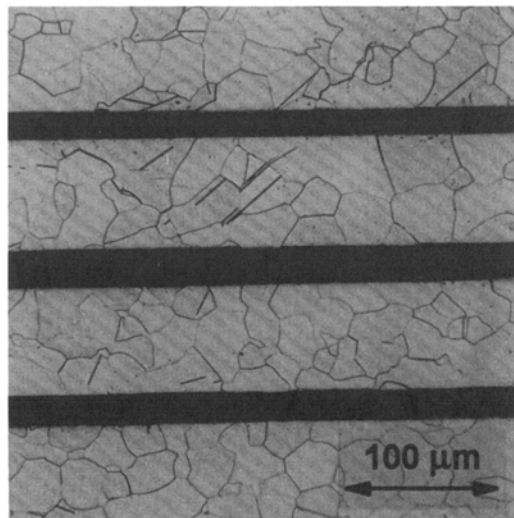
Fig. 7 Microstructures of typical rolled sheets made from Mo-47.5Re blended powder alloy. (a) 0.25 mm thick. (b) 0.13 mm thick. (c) 0.05 mm thick. All at 200×

Table 2 Data after long-time annealing of Mo-47.5Re

Sample No.	Annealing temperature, K	Grain size range, μm	Microhardness, HV	σ-phase
1	1873	10-50	295	Yes
2	1873 → 1473(a)	5-60	291	Yes
3	2073	30-390	301	None
4	2073 → 1473(a)	25-360	298	None

(a) Reheating to 1273 to 1673 K in 50 K intervals gave results identical to those of the 1473 K test

ally appear in stringers. Optical microscopy indicates higher precipitate density at the grain boundaries, but direct evidence of such preferential nucleation remains to be determined by further electron microscopy investigations. It may even prove that, in connection with recrystallization, new grains have been nucleated on σ-phase particles.

Figures 6(c) and (d) show clearly that small volume fractions of discontinuous σ-phase precipitates remain after cold
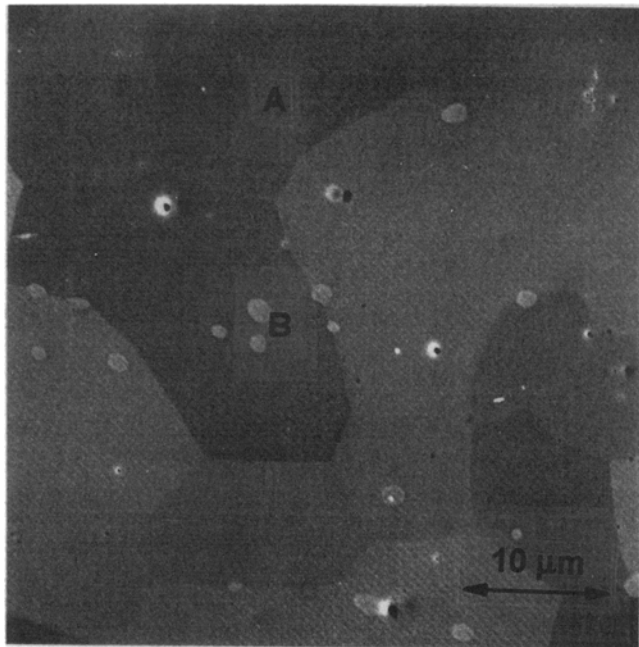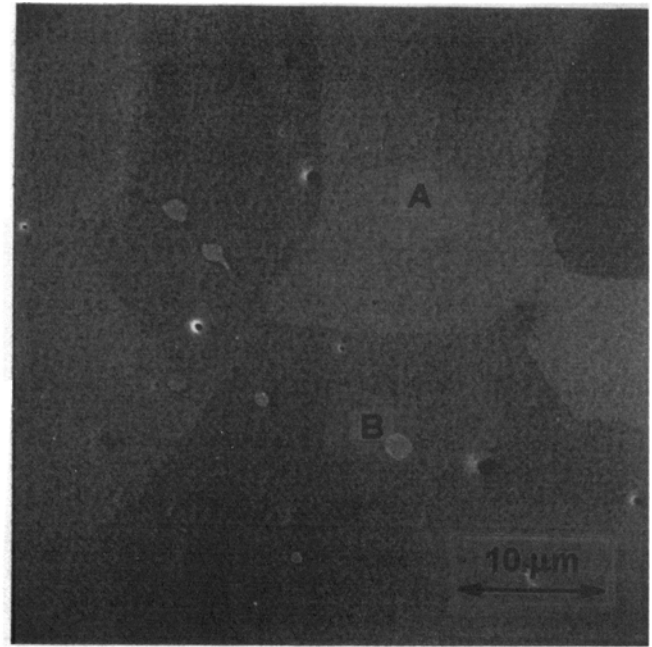
Fig. 8 Secondary electron images of Mo-47.5Re sample 1 (a) and sample 2 (b). Location A, matrix area; location B, area of σ-phase particle. 2000×

working to the final size of 0.5 mm. It appears that the σ-phase particles cannot be completely dissolved and diffused into the matrix despite the sufficiently high annealing temperature (i.e., 1873 K). This can be explained by the absence of substructures favoring pipe diffusion. However, during subsequent processing to a thickness of 0.050 to 0.025 mm, the structure will again become virtually free of σ-phase, as demonstrated on standard Mo-47Re products (Fig. 7a to c).

Mo-47.5Re sheet made from coated powder also shows some σ-phase after both hot and cold rolling. However, the quantity of precipitates is much smaller. It is also much easier to dissolve the σ-phase in subsequent process annealings. Such material is preferred when it will be used (or further fabricated) under conditions that may promote σ-phase formation.

Studies of the process of σ-phase formation in Mo-47.5Re alloys suggest that temperature and stress/strain conditions are the important driving forces for precipitation. These factors initiate formation of σ-phase during warm rolling. Subsequent cold-forming and annealing operations assist the nucleation of additional σ-phase particles. The main reason for this nucleation is the presence of large amounts of dislocations and twins in the bulk structure that promote pipe diffusion. The frequency of high-temperature annealing controls the slow dissolution and diffusion process of the σ-phase when the material is processed to thinner sizes.

3.2 Effects of High-Temperature Heating and Lower-Temperature Reheating with or without Intermediate Cold Working

The results of heat treatments of Mo-47.5Re at 1873 and 2073 K and reheating to 1273 to 1673 K for 25 ks are summarized in Table 2. The 1873 K temperature was not high enough

to dissolve all σ-phase during 1.8 ks. The 2073 K heat treatment resulted in a clean, σ-phase-free structure. Reheating to temperatures in the range of 1273 to 1673 K did not cause any σ-phase formation in the σ-phase-free sample, and no additional σ-phase was found in the material first heat treated at 1873 K. Figure 8 shows rounded to oval σ-phase particles. Figure 9 illustrates the EPMA analyses of the matrix and the particles. Comparison of the peaks in the matrix and the precipitates indicates 70% Re and 30% Mo in the σ-phase, which is to be expected. It can be concluded that reheating of stress-free samples with a supersaturated solution of rhenium in molybdenum in the temperature range of 1273 to 1673 K for 25 ks is not sufficient to cause σ-phase precipitation. A further proof is that no rhenium peaks caused by submicroscopic σ-phase could be found in the grain boundaries (Fig. 10).

Figure 11 shows a micrograph of Mo-47.5Re sheet annealed at 2073 K to dissolve all σ-phase and then rolled to 0.85 mm thickness. In the cold-worked material, σ-phase is not present. Annealing at 1273 to 1773 K results in σ-phase precipitation (difficult to illustrate by optical microscopy before recrystallizing), peaking at about 1573 K (Fig. 11b and c). When reheated to 1873 K, the σ-phase has almost completely dissolved (Fig. 11d). These results show clearly that σ-phase will be precipitated in the temperature region from 1273 to 1773 K if the material has been cold worked. To activate σ-phase formation it can be assumed that the following conditions must prevail: temperature on the order of 1273 to 1773 K and stresses high enough to create substructures (dislocations, twin lines, etc.) that promote pipe diffusion. Bulk diffusion in stress-free materials is not enough to cause σ-phase formation within reasonable periods of time.
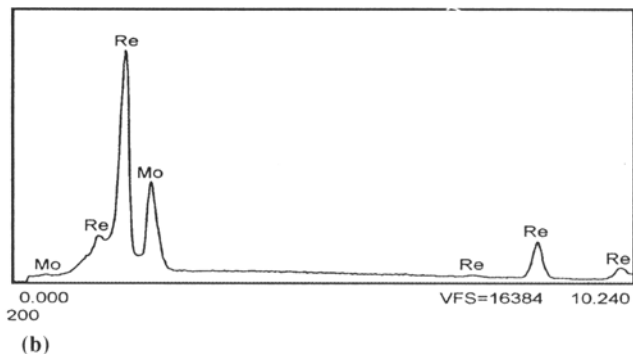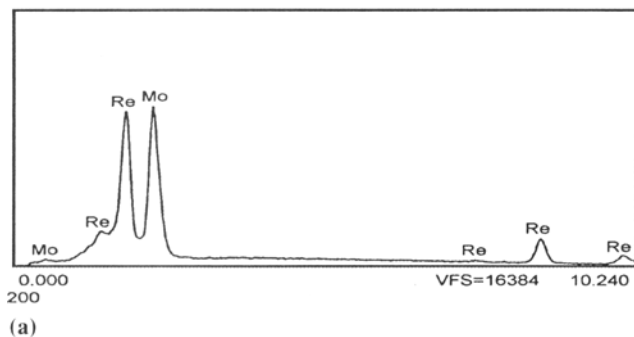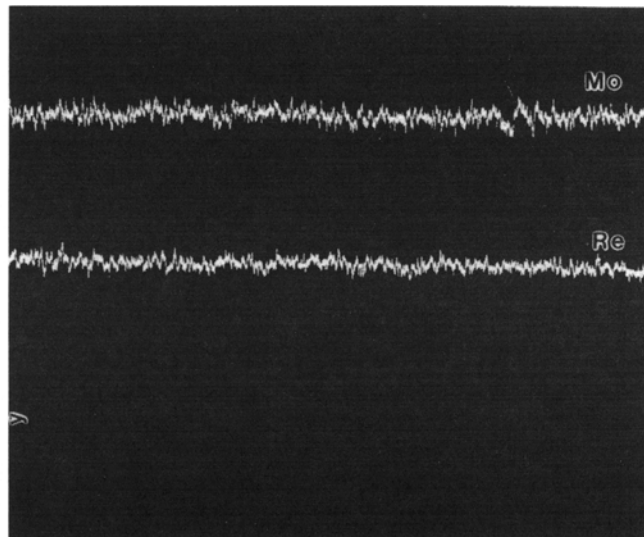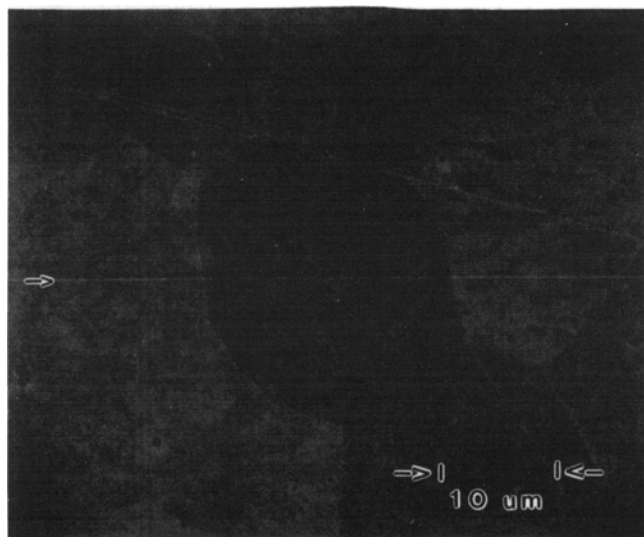
Fig. 9   X-ray EPMA analyses of sample 2. (a) Location A. (b) Location B

(a)   (b)

Fig. 10   (a) Secondary electron image of Mo-47.5Re sample 2. (b) Line profile across two grain boundaries. Both at 2000×

3.3   Recrystallization Study

In connection with the high-temperature annealing and re-heating investigation, the material heat treated at 2073 K was also used to study the effects of stress relieving and recrystallization. Microhardness values versus annealing temperature are shown in Fig. 12 and compared with results from previous studies of sheet and tubing conducted at Sandvik. The heavier sheet (0.85 mm) was fully recrystallized at 1573 K and the reference materials at 1673 K. Average grain size was 22 to 32 μm after recrystallizing and increased to a maximum of 90 μm after annealing at 1873 K. The results of this investigation agree well with previously published results and make it possible to select proper heat-treating parameters for Mo-47.5Re materials.

4. Conclusions

- Molybdenum alloys containing 41 or 44.5% Re are virtually free of σ-phase under all conditions studied. As the rhenium effect (improved low-temperature ductility and high-temperature strength) reaches a maximum at the solu-

bility limit, the Mo-44.5Re alloy should be the better of the two alloys.

- Mo-47.5Re alloys contain σ-phase when produced according to normal procedures. Coated powder yields a more homogeneous structure (less σ-phase) than regular powder blends.

- The precipitation of σ-phase in Mo-47.5Re is activated by a combination of temperature and stress/strain. The presence of substructures promoting pipe diffusion is probably a prerequisite for σ-phase formation.

- There is no difference in hardness for alloys containing between 41 and 47.5% Re using normal production parameters. There is no sign of precipitation hardening in the alloys containing small amounts of σ-phase.

- The chemical analysis of the matrix of Mo-47.5Re with a small amount of σ-phase seems to be very close to that of the Mo-44.5Re alloy. It can hence be assumed that these two alloys have very similar low- and high-temperature properties.

- The supersaturation with rhenium in Mo-47.5Re annealed at 2073 K to dissolve all σ-phase does not create a driving force high enough to reprecipitate σ-phase within reason-
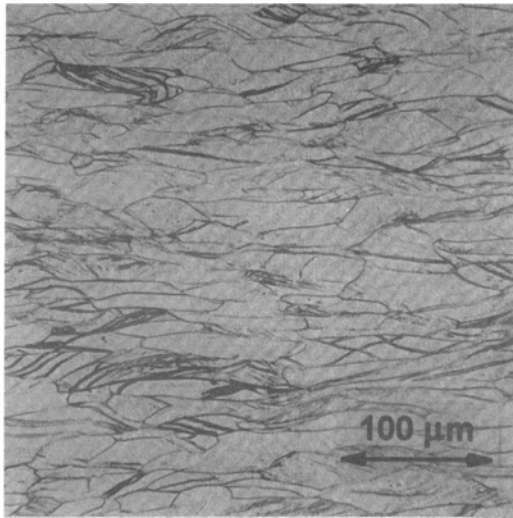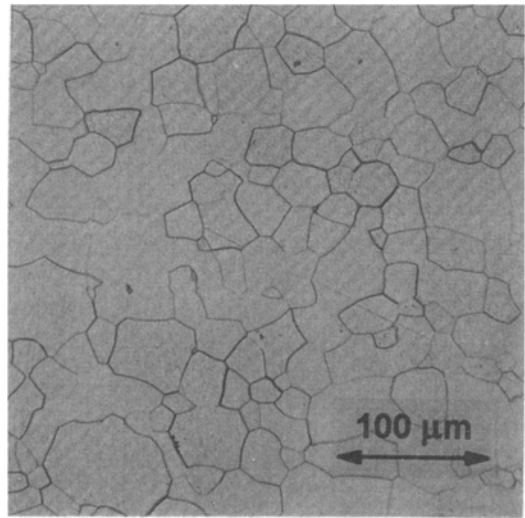
(a)
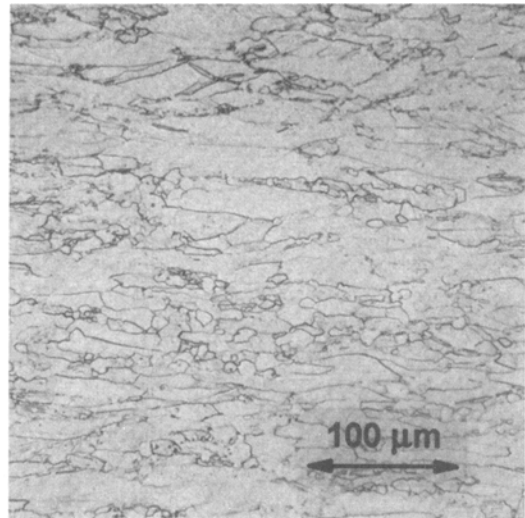
(b)
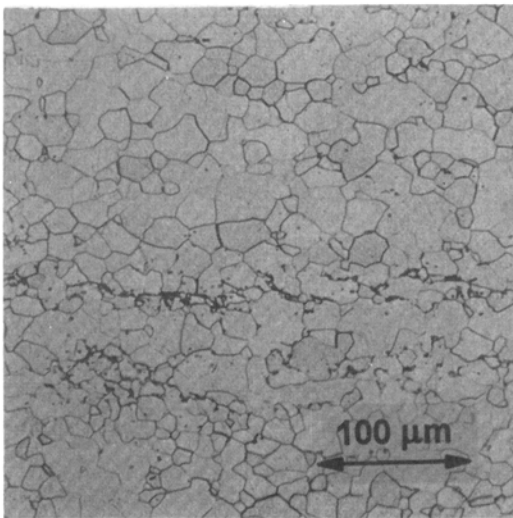
(c)

(d)

Fig. 11   Microstructure of Mo-47.5Re blended powder sheet, 0.85 mm thick, before and after 3.6 ks annealing in hydrogen. (a) As cold rolled. (b) As annealed at 1473 K. (c) As annealed at 1573 K. (d) As annealed at 1873 K. All at 200×
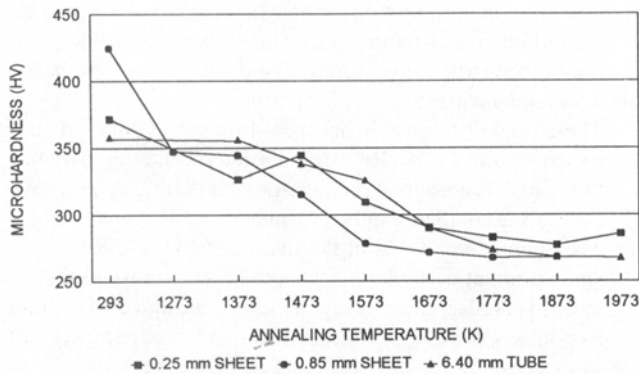

Fig. 12   Microhardness of Mo-47.5Re blended powder alloy as a function of annealing temperature. Time of exposure: 3.6 ks for 0.85 mm sheet; 0.9 ks for 0.25 mm sheet and 6.40 mm tube

able periods of time at temperatures from 1273 to 1673 K. After cold working, σ-phase will be precipitated in the same temperature interval.

- The morphology of σ-phase in Mo-47.5Re alloys using conventional production parameters is such that no embrittlement can be expected. The σ-phase appears as well-rounded particles that are not concentrated at the grain boundaries.

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