Molybdenum in Nb-C Alloys

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The effects of molybdenum alloying additions to niobium on the carbide phases and their precipitation behavior were investigated. The experimental alloys included Nb-0.1C, Nb-15Mo-0.1C, and Nb-30Mo-0.1C. After selected heat treatments the microstructural changes were determined by metallography and the carbide phases were extracted and identified by X-ray diffraction and chemical analysis. The results are essentially in agreement with recent phase diagram determinations. Additions of 30 wt pct Mo appears to slightly increase the solubility of carbon in niobium at temperatures around 1650°C. The solubility of molybdenum in Nb_2C is very small. Discontinuous precipitation of β-Nb_2C was found to occur in the Nb-30Mo-0.1C alloy during annealing at 1200°C. The important, overall effect of molybdenum in Nb-C alloys is to decrease the rate of niobium carbide precipitation so that appreciable carbon supersaturation can be achieved even after comparatively slow furnace cooling.

DISPERSED carbide phases are known to improve the high temperature strength properties of niobium-base alloys.[1-9] The degree of strength improvement depends largely on the distribution, morphology, and stability of the dispersed carbide phases. Among the carbide phases commonly observed in niobium alloys, the monocarbides of zirconium and hafnium are most attractive while the niobium-rich base-metal carbides are ineffective, since the latter usually either occur as coarse particles or coagulate rapidly. It has recently become apparent, however, that the degree of dispersion of the zirconium or hafnium carbide phases is determined to a large extent by the type and morphology of niobium-rich base-metal carbides[10] which are usually present prior to the formation of the dispersed, stable carbide phase during the required heat treatment cycle. For the development of improved carbide-dispersion strengthened niobium alloys it appears, therefore, vital to consider the influence of alloying additions on the properties of not only the dispersed zirconium- or hafnium-rich monocarbides but also of the niobium-rich base-metal carbide phases.

The base-metal carbide phases in niobium alloys with approximately 1 mol pct ZrC or HfC are of the hexagonal Nb_2C type[1-5] and of the carbon-richer ϵ type[10,11] with a yet unknown lattice structure resembling ζ-Ta carbide.[12] A detailed analysis of the precipitation behavior of niobium alloy D-43 has revealed that the hexagonal Nb_2C-type phase tends to form preferentially at grain boundaries and other lattice defects at temperatures above 1200°C whereas the ϵ-type carbide forms apparently in a more homogeneous mode,[10] as indicated by the Widmannstätten arrangement, at temperatures as low as 500° to 600°C. A carbide phase similar to the ϵ type has also been observed in niobium with less than approximately 1 at. pct C in an early investigation of the Nb-C system.[13] These base-metal carbide phases form rapidly during fast radiation cooling and moderate quenching from high annealing temperatures[10,11] and thereby reduce the available carbon supersaturation for the precipitation of the more stable monometal carbides.

A more homogeneous precipitation of stable monocarbides can probably be achieved if alloying elements are added to niobium which suppress or slow down the formation of nonequilibrium niobium carbide phases. Experience with current niobium-base alloys shows that the alloying elements—usually tungsten and zirconium or hafnium—do not act as sufficient inhibitors. However, scant evidence in the literature indicates that molybdenum additions to niobium may produce the desired inhibiting effect. Ostermann and Bollenrath found that oil quenching after solution annealing suppressed the formation of coarse niobium-rich carbides in alloy D-31 (Nb-10Mo-10Ti-0.1C) but not in alloy D-43 (Nb-10W-1Zr-0.1C) and microstructures of Nb-Mo-C alloys in Taylor and Doyle's report[14] indicate a significant influence of molybdenum on the precipitation behavior of niobium carbides. Recently determined phase equilibria in the Nb-Mo-C system[14-16] reveal a very limited solubility of Mo_2C in Nb_2C. Rudy *et al.*[16] obtained solubility values of approximately 3, 4, and 5 mole pct Mo_2C in Nb_2C at 1500°, 1900°, and 2200°C, respectively; the niobium-rich subcarbide is in equilibrium with metal-rich solid solutions containing up to approximately 30 to 40 at. pct Mo. Since, according to recent results of Rudy *et al.*[16], molybdenum continuously increases the solidus temperature of niobium and is a potent high temperature solid solution strengthener,[17-19] it is of considerable interest to determine the effects of molybdenum additions on the precipitation phenomena of carbides in ternary Nb-Mo-C alloys.

EXPERIMENTAL PROCEDURES

Three alloys were prepared with the following nominal compositions (in weight percent):

Nb-0.1C
Nb-15Mo-0.1C
Nb-30Mo-0.1C

The alloys were melted from chips and cuttings into 50 g buttons in a tungsten arc furnace under an environment of $\frac{1}{2}$ atm of purified argon. Prior to melting

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the alloys, a lump of titanium was melted to getter residual gases. The buttons were remelted three times to ensure homogeneity. The chips and cuttings were taken from commercially pure molybdenum, electron-beam melted niobium, and from a Nb-0.42 pct C alloy produced from the same high purity niobium and spectrographic quality graphite by consumable electrode arc-melting. The chemical analysis of the electron-beam melted niobium was as follows:

Element	C	N	O	H	Ta	W	Mo	Fe, Ni, Co, Al, Zr, Ti
	<5	<7	<14	<5	<200	<50	<40	<20 ppm each

The as-melted alloys were found to have the following compositions (wt pct):

Nb-0.116 pct C
Nb-14.70 pct Mo-0.111 pct C
Nb-26.85 pct Mo-0.099 pct C

Heat treatments were performed in a vacuum furnace equipped with a resistance-heated tungsten-mesh element and a molten tin bath container, into which the sample could be dropped from the hot zone by an externally operated release mechanism. Pressure was kept below 1 × 10^{-5} torr during heat-up and at temperature. Except for anneals at 1200°C, samples were not wrapped in protective tantalum foil to avoid differences in interstitial contents between tin-quenched and furnace-cooled samples. While wrapping of samples for heat treatment is useful for reducing contamination and decarburization, it cannot be used in tin-quenching experiments. Temperature was monitored by a W-WRe thermocouple attached to the sample or positioned close to it, if the sample was to be quenched. The temperature of the tin quenching bath was approximately 250°C.

Metallographic preparation, hardness testing, phase extraction, and X-ray diffraction procedures have already been described elsewhere.[10,11]

RESULTS

The alloy buttons were sectioned into samples of approximately 5 g and subjected to one of the following vacuum heat treatments:

A) 1750°C for 1 hr + tin quench
B) 1650°C for 1 hr + tin quench
C) 1750°C for 1 hr + furnace cool
D) 1750°C for 1 hr + furnace cool followed by 1200°C for 24 hr + furnace cool

The radiation cooling in the furnace was monitored by a W-WRe thermocouple attached to the samples and is illustrated in Fig. 1. The initial cooling rate from 1750° to 1350°C averages to 400°C per min. The quenching rate is estimated to be about an order of magnitude higher. Previous work has shown that quenching in liquid tin from high temperatures effectively prevents the formation of coarse niobium carbides which are commonly observed after furnace cooling.[10]

Room temperature hardness data taken after heat treatments are illustrated in Fig. 2. The large influence of annealing temperature and cooling rate on the hardness of each individual alloy can very likely be ascribed to the state of carbon after each heat treatment. For each alloy the lowest hardness was measured after the 1750°C anneal followed by furnace cooling and additional annealing at 1200°C. These low hardness values are probably influenced very little by dissolved carbon and coagulated carbide phases. The increasing hardness with increasing molybdenum content is then due to the solid solution hardening effect of molybdenum. The high hardness values exhib-

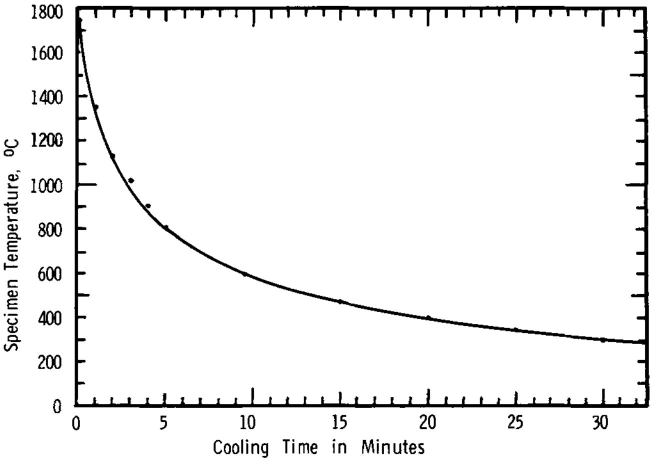


Fig. 1—Cooling curve for specimens annealed at 1750°C (furnace cooled). Total weight of specimens is approximately 30 g.

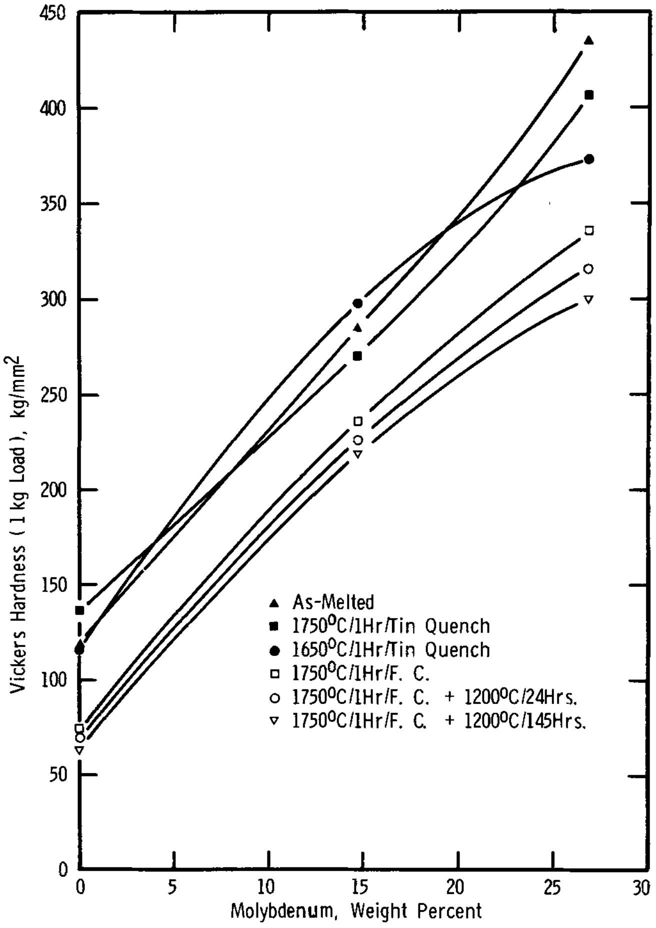
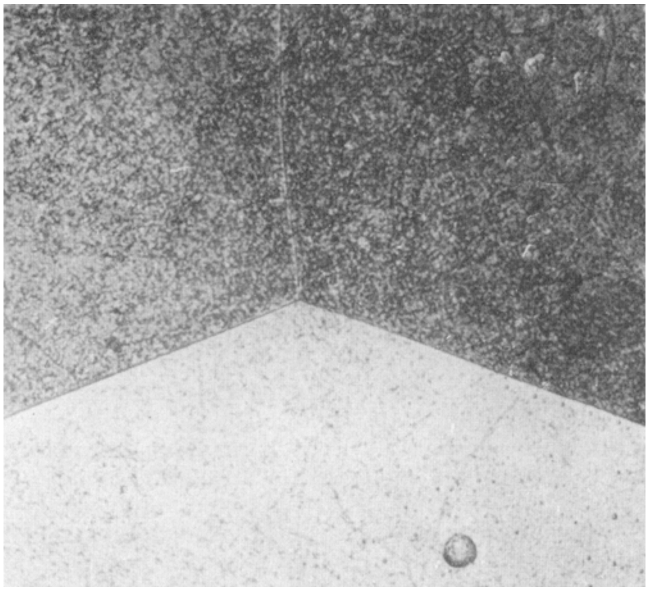


Fig. 2—Hardness of Nb-Mo-0.1C alloys vs molybdenum content in various heat treated conditions.

ited by the as-melted and the quenched samples cannot be accounted for entirely by hardening due to interstitially dissolved carbon, but reflect precipitation or dispersion hardening due to the carbide phases formed during cooling.

Typical microstructures of the heat treated samples are shown in Figs. 3, 4, and 5, for each alloy respectively. Precipitate phases are seen in all microstructures of the binary alloy Nb-0.1C, Fig. 3. The sample quenched after annealing at 1750°C, Fig. 3(*a*), contains a fine and uniformly dispersed carbide phase. In contrast to the quenched sample, large carbide particles can be seen in the furnace-cooled samples, although there is also a minor amount of acicular-shaped precipitate phase, which appears to decrease upon annealing at 1200°C, Figs. 3(*b*) and (*c*). As shown later, the cooling method not only controls the size and distribution, but also the type and lattice structure of the carbide phases present after annealing at 1750°C, Table I. Precipitation of the carbide phases shown in Figs. 3(*a*) and (*b*) must, therefore, have occurred during cooling or quenching, *i.e.* complete solid solution existed during annealing at 1750°C. After annealing at 1650°C a fine, dispersed carbide phase, which is essentially identical to the one in Fig. 3(*a*), again appears to precipitate during quenching in the liquid tin bath. However, a small amount of coarse, primary carbides was also observed at some grain boundaries in this sample, indicating that the carbon content of the sample is slightly in excess of the carbon solubility of niobium at 1650°C. Since decarburization of niobium can easily occur at high annealing temperatures,[10,11] the heat treated samples were chemically analyzed for their interstitial elements:

Heat Treatment	Carbon	Oxygen	Nitrogen
1750°C/1 hr/tin quench	0.083 pct	12 ppm	31 ppm
1650°C/1 hr/tin quench	0.096 pct	–	–



(*a*)

Fig. 3—Effects of heat treatment on microstructure of alloy Nb-0.1C, (*a*) 1750°C for 1 hr, tin quenched, (*b*) 1750°C for 1 hr, furnace cooled, and (*c*) 1750°C for 1 hr, furnace cooled + 1200°C for 24 hr. Magnification 575 times.

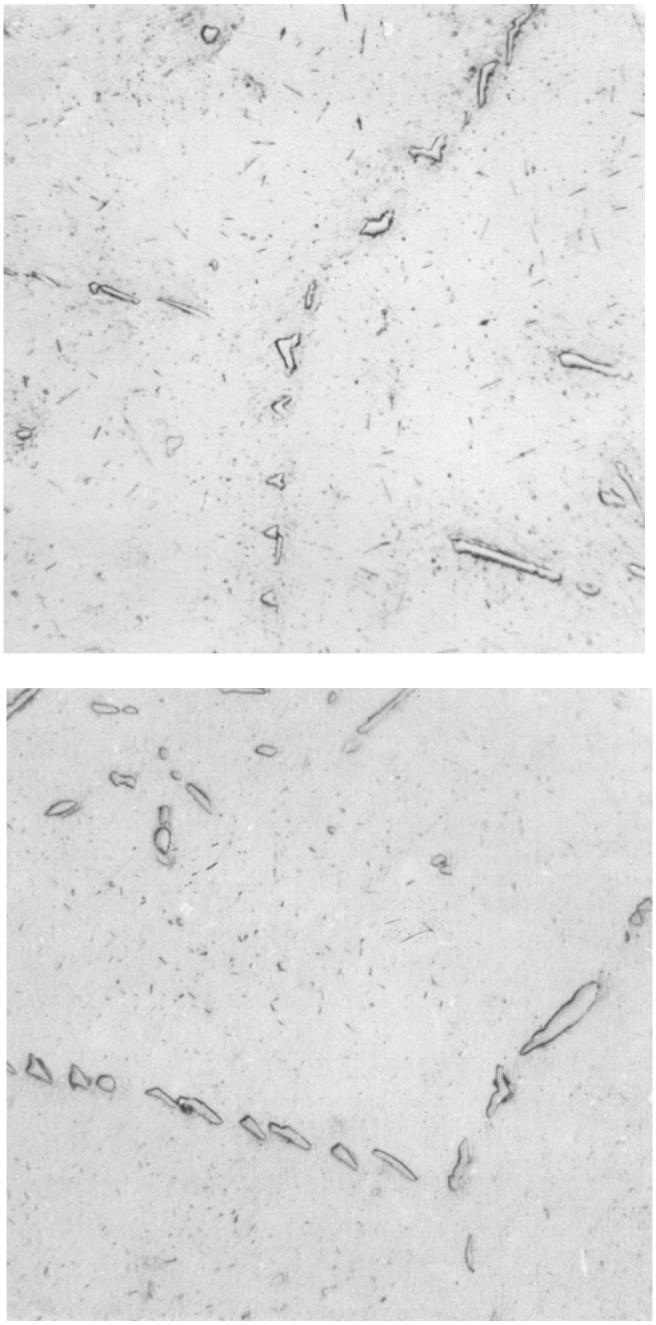
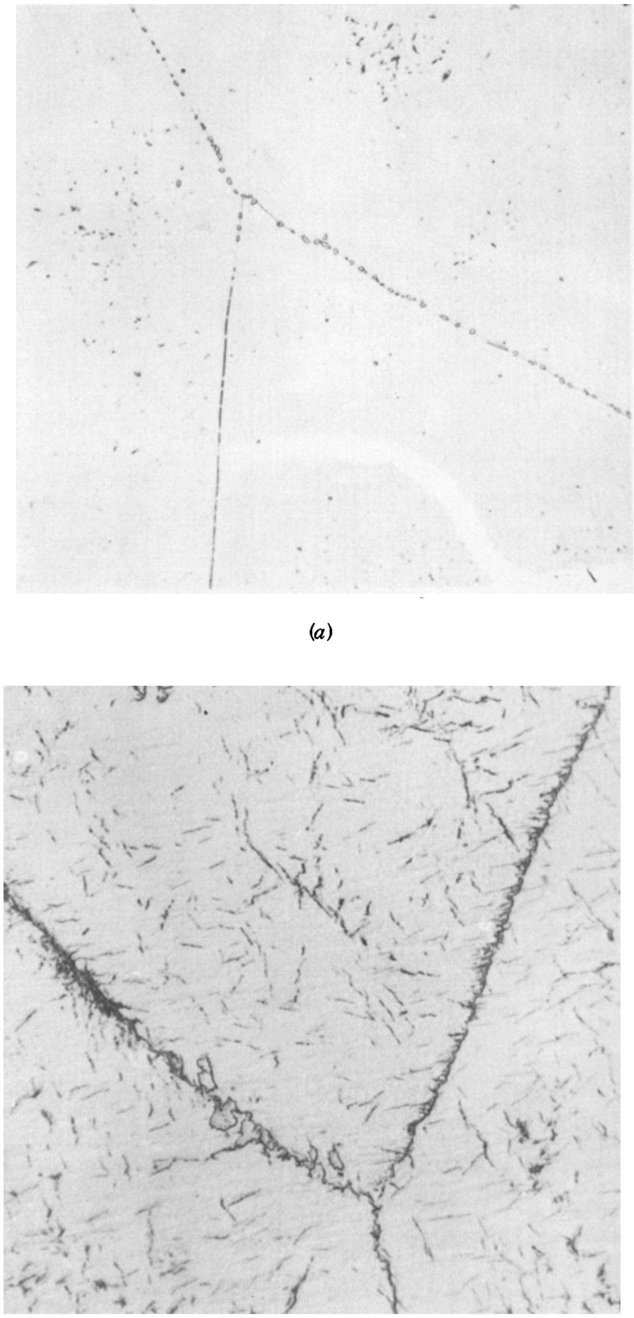


Fig. 3—Continued. (*c*)

It is estimated that the excess carbon in the form of primary carbides at 1650°C amounts to about 50 to 100 ppm. Since the chemical carbon analysis is accurate within ±30 ppm, the solubility of carbon in niobium at 1650°C is estimated to be 0.089 ± 0.005 wt pct.

When the Nb-15Mo-0.1C alloy is annealed at 1750°C and tin quenched, the structure appears to be single phase. A small amount of second-phase particles is observed after annealing at 1650°C and tin quenching, Fig. 4(*a*). Chemical analysis after heat treatment gave the following composition of this alloy:

Heat Treatment	Carbon	Oxygen	Nitrogen
1750°C/1 hr/tin quench	0.087 pct	37 ppm	26 ppm
1650°C/1 hr/tin quench	0.100 pct	–	–



(a)

(b)

Fig. 4—Effects of heat treatment on microstructure of alloy Nb-15Mo-0.1C (a) 1650°C for 1 hr, tin quenched, (b) 1750°C for 1 hr, furnace cooled, and (c) 1750°C for 1 hr, furnace cooled + 1200°C for 24 hr. Magnification 560 times.

Taking into account approximately 100 to 150 ppm excess carbon contained in the primary carbides, the solubility of carbon in Nb-15Mo (nominal) at 1650°C is 0.088 ± 0.005 pct according to these results.

The Nb-30Mo-0.1C alloy was found to be of single phase after annealing and quenching both from 1750° and 1650°C, Fig. 5(a). The interstitial alloy content was determined as follows:

Heat Treatment	Carbon	Oxygen	Nitrogen
1750°C/1 hr/tin quench	0.079 pct	58 ppm	42 ppm
1650°C/1 hr/tin quench	0.089 pct	–	–

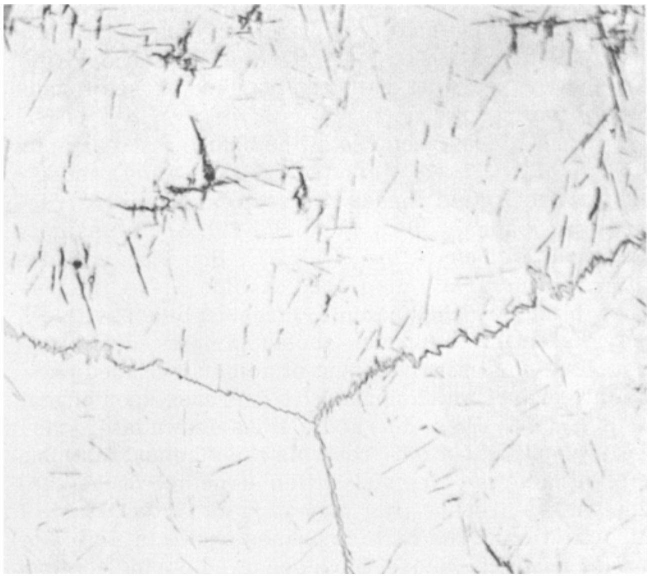


Fig. 4—Continued. (c)

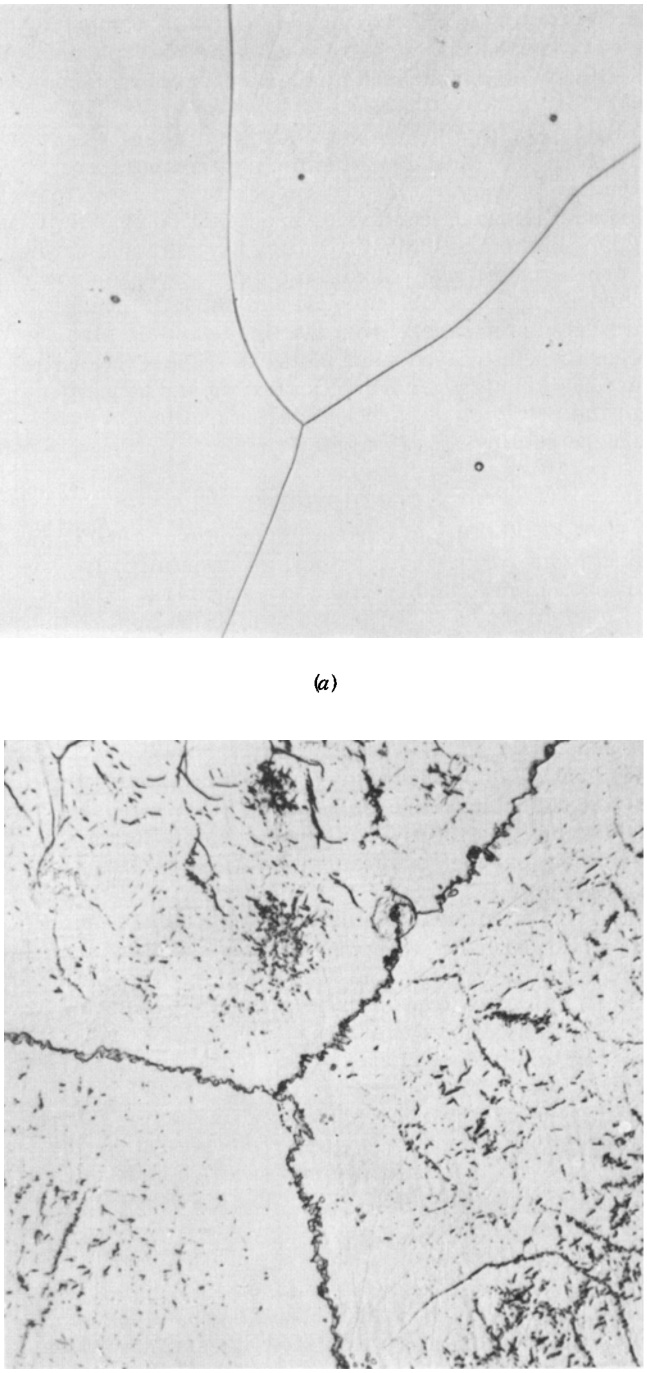
From these results, the carbon solubility in Nb-30Mo (nominal) at 1650°C appears to be in excess of 0.089 ± 0.003 wt pct.

A comparison of the microstructure of the Nb-15Mo-0.1C and Nb-30Mo-0.1C alloys after annealing at 1750°C and furnace cooling, Figs. 4(b) and 5(b), with the corresponding microstructures of the binary Nb-0.1C alloy, Fig. 3(b), reveals significant differences. While heavy precipitation at grain boundaries still persists, the carbide phase is less coagulated the higher the molybdenum content of the alloy. Also, the intragranular precipitates have an acicular shape, their size diminishes with increasing molybdenum content and they prefer to form at subboundaries and in residual segregated areas.

Additional annealing at 1200°C causes apparent growth of the acicular phase in the Nb-15Mo-0.1C alloy, Fig. 4(c). Although it can also be observed occasionally in the Nb-15Mo-0.1C alloy, discontinuous precipitation becomes very pronounced in the Nb-30Mo-0.1C alloy at 1200°C as shown in Fig. 5(c). Lamellar growth of depleted matrix and precipitate phase into the supersaturated matrix originates at grain-boundaries and subgrain boundaries, the old and new phase regions being clearly separated by high-angle boundaries. Extended annealing at 1200°C does not appear to cause any further growth except for some coagulation of the carbide lamellae.

The carbide phases observed in the microstructures were identified by X-ray diffraction as shown in Table I. The fine precipitate phase extracted from the binary Nb-0.1C alloy after annealing at 1750°C and tin-quenching produced the characteristic diffraction pattern of the ϵ carbide, which was identified originally in the niobium alloy D-43.[11] The carbon content of this phase in the binary alloy was chemically analyzed and is 7.48 wt pct or 38.47 at. pct. A complete chemical analysis of this phase was not made, but it can be assumed from earlier work[1,11] that the residue contains approximately 5 to 6 wt pct O which is apparently picked up in the extraction process. The carbon content of 7.48 wt pct, therefore, should be regarded as the lower limit and may be as high as 7.95 wt pct or 40.0 at. pct, which corresponds to the stoichiometric composition of Nb_3C_2.

The carbide phases extracted from Nb-0.1C samples after annealing at 1750°C and furnace cooling and after the additional anneal at 1200°C proved to be α-Nb_2C with the characteristic diffraction line doublets. The α-Nb_2C phase was apparently observed simultaneously by Yvon, Nowotny, and Kieffer,[21] Rudy and Brukl,[20] and Bowman *et al.*, Ref. cited in Ref 21. It exists at temperatures below 1230°C[20] and its structure is an orthorhombic distortion of the hexagonal L′3 cell. The distortion is thought to be the result of an ordered arrangement of carbon atoms in the lattice. Neutron diffraction analysis by Yvon *et al.*[21] gave the following lattice parameters: a = 10.920, b = 4.974, and c = 3.090Å. Indexing the α-Nb_2C phase in the Nb-0.1C alloy in this manner resulted in excellent agreement of the lattice parameters, see Table I.



(a)

(b)

Fig. 5—Effects of heat treatment on microstructure of alloy Nb-30Mo-0.1C, (a) 1650°C for 1 hr, tin quenched, (b) 1750°C for 1 hr, furnace cooled, and (c) 1750°C for 1 hr, furnace cooled + 1200°C for 24 hr. Magnification 560 times.

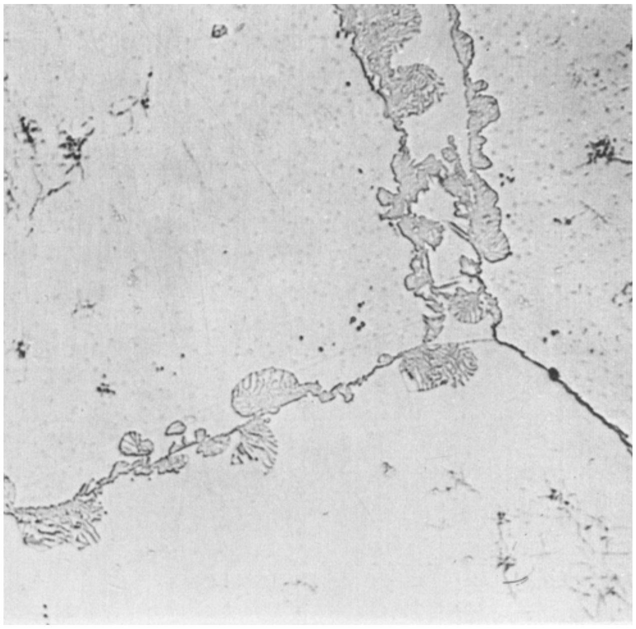


Fig. 5—Continued. (c)

After furnace cooling from 1750°C both ternary alloys contained hexagonal β-Nb_2C. The lattice parameters (a = 3.128; c = 4.970; c/a = 1.589) are virtually identical for the carbide phases of both alloys. The Nb-15Mo-0.1C alloy contained a minor amount of ϵ carbide which was absent in the Nb-30Mo-0.1C alloy. Annealing at 1200°C for 24 hr caused the β-Nb_2C and ϵ carbide phases in the Nb-15Mo-0.1C alloy to transform into α-Nb_2C. This transformation was not observed in the Nb-30Mo-0.1C alloy, although the diffuse appearance of the high-angle diffraction lines may indicate the early stage of the transformation. In the 1200°C annealed condition the carbide phases were analyzed for molybdenum content. The Nb_2C phases of the Nb-15Mo-0.1C and Nb-30Mo-0.1C alloys contained 1.41 and 2.83 wt pct Mo, respectively.

DISCUSSION

The Nb-C system has been the subject of several investigations. With respect to the determination of carbon solubility in niobium, the works of Elliott,[22] Kimura and Sasaki,[23] and Gebhardt, Fromm, and Roy[24] are particularly noteworthy. According to the results of these investigations, a maximum of 0.09 wt pct (0.70 at. pct) C dissolves at 1750° ± 25°C. This temperature is about 100°C higher than determined in the present investigation. The metallographic evaluation employed here may overestimate the solubility of carbon slightly but not to the extent necessary to comply with the published data. Approximately 0.09 wt pct C dissolves in alloy D-43 (Nb-10W-1Zr-0.1C) at 1675° ± 25°C,[11] although the solubility should be decreased by the alloy-

Table I. Results of Phase Identification by X-Ray Diffraction

Alloy and Heat Treatment	Phases Identified	Relative Amount	Lattice Structure	Lattice Parameter, Å
Nb-0.1C·				
1750°C/1 hr/tin quenched	ε("Nb_3C_2")	100	not det.	not det.
1750°C/1 hr/furnace cooled	α-Nb_2C	100	o'rhombic	a = 10.92 b = 4.97 c = 3.10
1750°C/1 hr/furnace cooled + 1200°C/24 hr/furnace cooled	α-Nb_2C	100	o'rhombic	a = 10.912 b = 4.972 c = 3 100
Nb-15Mo-0.1C:				
1750°C/1 hr/furnace cooled	ε("Nb_3C_2")	30	not det.	not det.
	β-Nb_2C	70	hexagonal	a = 3.128 c = 4.970
1750°C/1 hr/furnace cooled + 1200°C/24 hr/furnace cooled	α-Nb_2C	100	o'rhombic	a = 10.94 b = 4.971 c = 3.105
Nb-30Mo-0.1C				
1750°C/1 hr/furnace cooled	β-Nb_2C	100	hexagonal	a = 3.128 b = 4.971
1750°C/1 hr/furnace cooled + 1200°C/24 hr/furnace cooled	β-Nb_2C	100	hexagonal	a = 3.128 c = 4.971

ing elements in this case. It is possible that the carburizing method used by Elliott and Gebhardt *et al.* will somewhat underestimate the solubility by not achieving complete equilibrium concentrations during carburization.

The additions of 15 and 30 wt pct Mo to niobium appear to have only a small effect on the carbon solubility, although the data indicate a moderate solubility increase in the Nb-30Mo alloy. These results disagree with the findings of Taylor and Doyle[14] who estimate a solubility of less than 0.1 at. pct (0.013 wt pct) C at 2000°C in Nb-10.3 wt pct Mo from metallographic evidence. The estimate of low solubility is most likely caused by erroneously taking the observed carbide phase as the primary carbide existing at the equilibrating temperature.

The observation that the concentration of molybdenum in Nb_2C is only about 10 pct of the molybdenum concentration of the alloy is in good agreement with the results of Rudy *et al.*[16] Since there is no measureable difference between the lattice parameters of the hexagonal Nb_2C phases present after furnace cooling from 1750°C and after additional annealing at 1200°C, see Table I, it can be assumed that the molybdenum content of the carbide phases is nearly the same after both heat treatments. The precipitation of Nb_2C in niobium with molybdenum contents of 30 to 40 wt pct, therefore, must involve considerable bulk diffusion of the metallic constituents in comparison with Nb_2C precipitation in pure niobium or in alloys where the concentration change of metallic elements between carbide and matrix is small.

The experimental results provide some qualitative evidence for the effects of molybdenum on the precipitation behavior of the niobium carbide phases. The rate of ε-carbide precipitation seems to decrease with increasing molybdenum content. The precipitate particles of the ε phase can be readily observed in the microstructure of the Nb-0.1C alloy after tin-quenching from around 1750°C, whereas only the high hardness of the molybdenum-bearing alloys after tin-quenching indicates the presence of carbide precipitation. Similarly, the ε phase appears after furnace cooling in the Nb-15Mo-0.1C alloy but not in the Nb-30Mo-0.1C alloy. The formation of ε carbide during cooling, which commonly occurs in a much lower temperature range than does the Nb_2C phase,[10] is effectively suppressed by the addition of molybdenum.

With regard to the Nb_2C phases, molybdenum appears to retard the transition from the high temperature β structure of Nb_2C to its low temperature α modification. The additional, discontinuous precipitation of β-Nb_2C at 1200°C in the Nb-30Mo-0.1C alloy may also indicate a decrease of the α-β transition temperature. A growth-inhibiting influence of molybdenum on Nb_2C precipitates is also apparent from the finer particle size. In general then, molybdenum additions to Nb-C alloys reduce the kinetics of carbide formation and hence permit the retention of carbon supersaturation during furnace cooling.

CONCLUSIONS

1) The solubility of carbon in niobium at 1650°C was found to be 0.089 ± 0.005 wt pct, which is slightly larger than reported by other investigators.

2) Additions of 30 wt pct Mo to niobium appear to increase slightly the carbon solubility at 1650°C.

3) The Nb_2C phases in the Nb-15Mo-0.1C and Nb-30Mo-0.1C alloys at 1200°C were found to contain 1.4 and 2.8 wt pct Mo, respectively.

4) The rates of precipitation of niobium carbides from solid solution are decreased considerably by molybdenum additions. Thus it is possible to achieve significant carbon supersaturation with practical cooling rates.

5) A classical example of discontinuous precipitation was observed in Nb-30Mo-0.1C after annealing at 1200°C.

6) The predominant carbide phases precipitating from solid solution were the orthorhombic α-Nb_2C in Nb-0.1C and the hexagonal β-$(Nb, Mo)_2C$ in the ternary alloys. The ε-(Nb_3C_2) carbide was only found in the binary alloy after tin-quenching and in the Nb-15Mo-0.1C alloy after furnace cooling from 1750°C. Orthorhombic α-Nb_2C formed during annealing at 1200°C for 24 hr in the Nb-15Mo-0.1C alloy but not in the Nb-30Mo-0.1C alloy.

7) It is expected that additions of 15 to 30 wt pct Mo to Nb-Zr (or Hf)-C alloys will result in precipitation characteristics of the stable carbide phases that are more desirable than are exhibited by current niobium-base alloys.

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