

## TEMPERATURE DEPENDENCE OF THE HARDNESS OF IRON-MOLYBDENUM AND NICKEL-MOLYBDENUM ALLOYS

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This paper communicates the results of short and long time hardness measurements on iron-molybdenum and nickel-molybdenum alloys.

Materials and Experimental Methods. The alloys were prepared in a 50 kg induction furnace. The iron-base alloys contained 4% Mo (No. 204) and 12% Mo (No. 212) and those on a nickel basis 7% Mo (No. 307) and 25% Mo (No. 325).

From the iron-molybdenum and nickel-molybdenum phase diagrams (Fig. 1), it is seen that alloys 204 and 307 are homogeneous over the whole temperature range, and do not undergo any transformations. Alloys 212 and 325, with higher molybdenum contents, are two-phase in the equilibrium condition, and are capable of precipitation hardening after quenching from the homogeneous region.

Alloys 204 and 307 were studied after vacuum annealing for 2 hours at 900°C (1650°F) followed by furnace cooling.

Specimens of alloy 212 were quenched from 1200°C (2190°F) in oil; those of alloy 325 in water from 900°C (1650°F). The aging of alloys 212 and 325 at different temperatures was followed by hardness measurements.

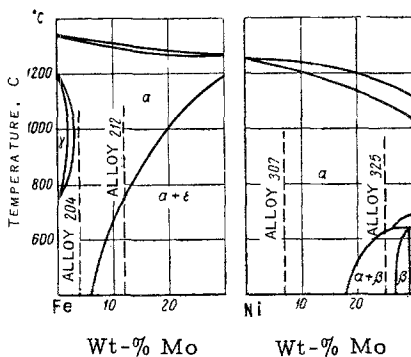


Fig. 1. Constitutional diagrams of iron-molybdenum (left) and nickel-molybdenum (right).

Fig. 2 shows the effect of aging on the microstructure of alloy 212. Seven hours' aging at 700°C (1290°F) (Fig. 2a) produces in the grains punctiform formations, which are apparently of the epsilon phase,  $Fe_3Mo_2$ . Increase of the aging temperature to 800°C (1470°F) somewhat, and to 900°C still more, coarsens these formations.

Note that aging at 800 and 900°C (1470, 1650°F) decreases the precipitation of epsilon phase at grain boundaries. In Figs. 2b and 2c, arrows indicate the zones around grain boundaries containing considerably smaller

amounts of these formations.

Hardness measurements were made with a six-spindle rig, [1,2] in which the specimens were heated in vacuum. The apparatus was modernized in 1957, and the originally manual control of the mechanisms for lowering and raising the indentors and loads was replaced by an automatic arrangement. Fig. 3 shows the improved apparatus, whose actuating mechanism 1 consists of an encased

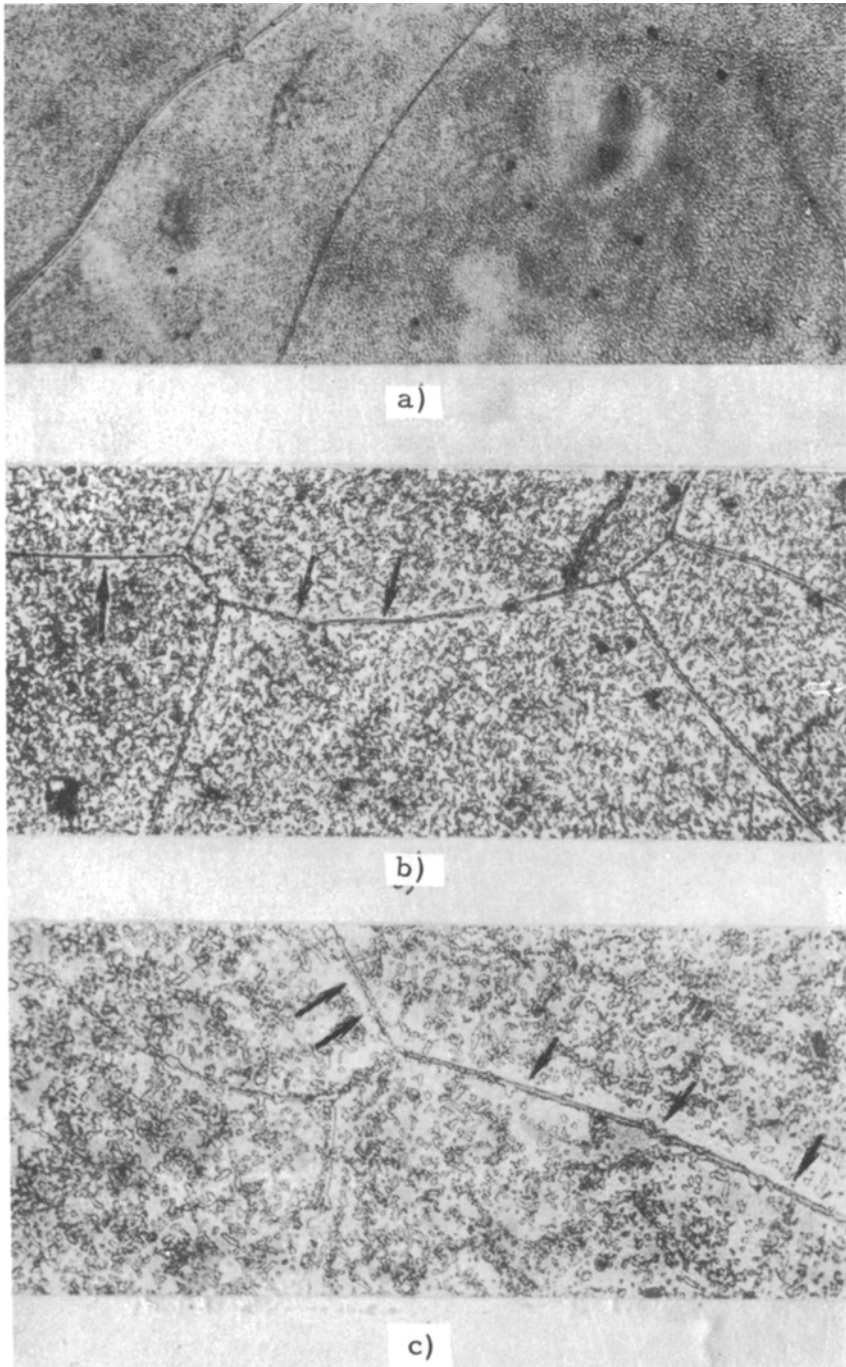


Fig. 2. Microstructure (of specimens) of alloy 212, aged at different temperatures,  $\times 500$ . a = seven hours at  $700^{\circ}\text{C}$  ( $1290^{\circ}\text{F}$ ); b =  $5\frac{1}{2}$  hours at  $800^{\circ}\text{C}$ , ( $1470^{\circ}\text{F}$ ); c = 5 hours at  $900^{\circ}\text{C}$  ( $1650^{\circ}\text{F}$ )

electric motor and reduction gear. A half turn of flange 2 displaces through a vacuum seal the indentors and loads 3, in the vacuum chamber.

The time of holding of the indentors on the specimens was controlled by a time relay. After a preset time, this relay switched on the electric motor of the actuating mechanism 1, which lifted the indenter and load.

The specimens were rotated inside the vacuum chamber with the knob 4 through  $6^\circ$  for the next indentation. The scale and pointer 5 show the displacement of the specimen from its original position.

The automated hardness measuring apparatus improved the reproducibility of hardness determinations in vacuum between 20 and 1300°C (68 and 2370°F).

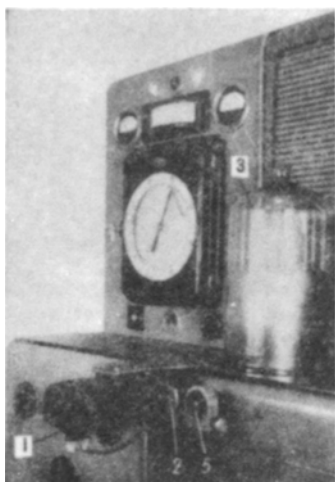


Figure 3

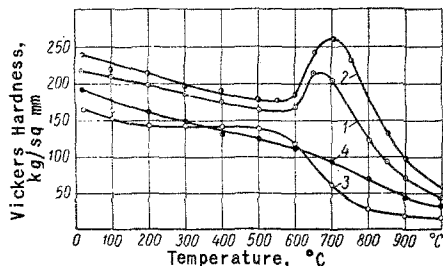


Fig. 4. Temperature dependence of the short-time hardnesses for all alloys at 20-1000°C in a vacuum of about  $10^{-5}$  mm Hg.

← Fig. 3. Apparatus for measuring indentation hardnesses in vacuum at up to 1300°C (2360°F).

Measurement of Short-Time Hardness of Fe-Mo and Ni-Mo Alloys at 20-1000°C (68-1830°F) in Vacuum.

In these experiments, the indenter was kept on the specimens for one minute. Fig. 4 shows the variation of short-time hardness of the four alloys (in Vickers units against test temperatures). The specimens of alloys 204 and 307 were all annealed whereas alloys 212 and 325 were solution treated.

Curve 1 shows a gradual decrease of hardness of 212 between 20 and 550°C (68-1020°F) which then rises to a maximum at 700°C (1290°F), subsequently falling at still higher temperatures. Curve 2 for alloy 325 is similar, its hardness gradually falling with temperature, rising again around 600°C (1110°F), reaching a maximum at 700°C and decreasing again beyond this point. The maxima at 700°C show that alloys 212 and 325 precipitation harden strongly at this temperature.

Curve 3 shows that there is hardly any change in alloy 204 up to 600°C, but between 600 and 800°C (1110-1470°F) there is a considerable softening, which levels off between 850 and 1000°C (1560 and 1830°F).

Curve 4, for alloy 307, shows a systematic fall of hardness with increase of temperature [3].

Kinetics of Aging of Alloys at 300-1000°C (570-1830°F).

Figs. 5 and 6 show the variations of alloys 212 and 325 during holding at constant temperature in vacuum. The curves for alloy 212 (Fig. 5) were plotted from isothermal experiments at 300-900°C (570-1650°F) and show a maximum hardness increase when aging occurs at 700°C. This is in accordance with the observations in the preceding section (Fig. 4).

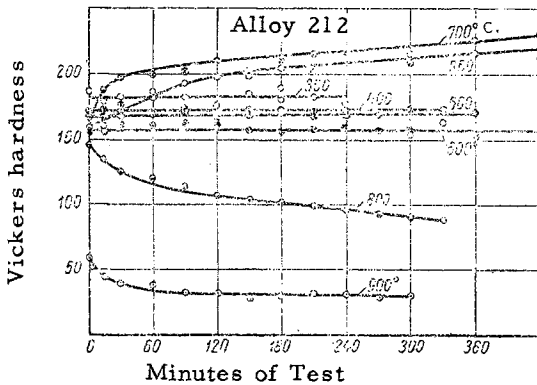


Fig. 5. Kinetics of aging alloy 212 (Fe-12% Mo) as reflected in its indentation hardness changes during isothermal vacuum heating.

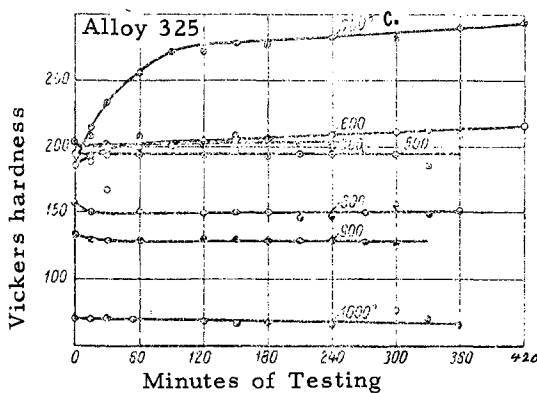


Fig. 6. As Fig. 5, for alloy 325 (Ni-25% Mo).

The curves for alloy 325 for 300-1000°C in Fig. 6 show that this alloy begins to age at 600°C (1110°F) with a slight increase in hardness. At 700°C the aging is faster and there is a greater increase of room temperature hardness after the experiment. The (short-time) hardnesses measured at 800-1000°C do not vary with time, nor are any changes in hardness observed after the test (at room temperature). Hence at these temperatures there is no aging of the alloy.

Temperature Dependence of 'Long-Time' Hardness. A comparison of the figures obtained from measurements on indentations made on the surface of a specimen at constant temperatures and loads, but different times of holding the indenter on the specimen under load (30, 300 and 3000 seconds) reveal that the alloy has a tendency to creep, the explanation of which is that indentation hardness characterizes the resistance of the specimen to plastic deformation, and a variation of the size of the indentation with time of loading can be compared with creep.

Fig. 7 shows the effect of time of isothermal holding on the long-time hardness of alloys 307 and 325. As the test temperature increases, unaged 307 alloy creeps at a constant rate at 500-800°C. At 900°C (1650°F) the creep rate, estimated by the difference in hardness for different times of keeping the indenter

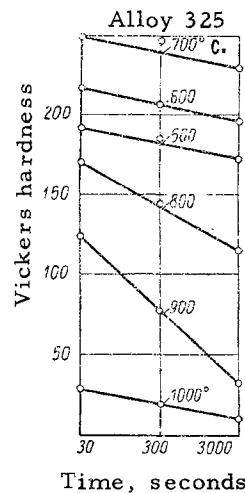
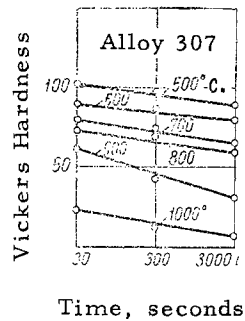


Fig. 7. Variation of long-time hardness.

under load, is higher. At 1000°C (1830°F) the slope of the hardness/time curve is less, showing a reduced tendency of the alloy to creep.

The observations on alloy 325 show that it softens (creeps) at approximately identical rates at 500-700°C. Note, however, that the hardnesses at 600 and 700°C (1110 and 1290°F) are higher than at 500°C (930°F), which can be explained by faster aging above 500°C (fastest of all at 700°C). When the test temperature is raised to 800°C (1470°F) the hardness is much lowered, and the difference in hardness between specimens held for 30 and 3000 seconds under load increases to 55 Vickers points. This proves that at 800°C, the aging of this alloy is terminated and it creeps more readily.

The difference in the 30 and 3000 seconds hardnesses at 900°C is 92.5 Vickers points, the corresponding figure for 1000°C being 19.5 points. It is interesting that the creep rate at 1000°C (Fig. 7) is less than at 800 and 900°C, and approximately the same as at 500, 600 and 700°C.

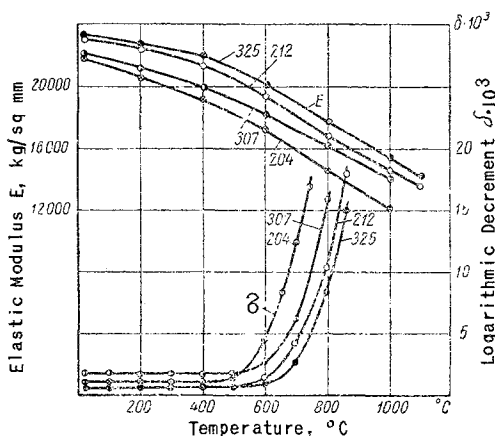


Fig. 8. Temperature variation of the elastic moduli and of internal friction (logarithmic decrement) of the alloys studied.

Fig. 8 shows temperature dependences of the basic parameters, the tests being made on a IMASh-6 apparatus [2, 4].

The alloys with the higher molybdenum contents have higher elastic moduli. For example, alloys 307 and 325 differ in elastic moduli at room temperature by about 1600 kg/sq. mm, and at 600°C by 2130 kg/sq. mm, but only by 1600 units again at 1000°C. The diagram shows that with similar molybdenum contents the elastic modulus is somewhat higher for nickel-base alloys.

The elastic moduli are hardly affected at all by quenching and aging, but the internal friction curve changes its course, this property being highly structure-sensitive.

A study of the deformation of these four alloys combined with an investigation of their microstructures at various stages of high temperature failure revealed a close correlation between strength properties and short and long time hardnesses.

#### SUMMARY

1. The effect of alloying iron and nickel with molybdenum has been studied, and it has been shown that the Mo concentration converting the alloys from one-phase to two-phase structures, hardens them over the whole temperature range studied.
2. Up to 700°C (1290°F) there is precipitation hardening in the two-phase region, which greatly increases hardness.
3. The long-time hardness of the nickel alloy with 25% molybdenum is higher at 600 and 700 than at 500°C (1110, 1290 and 930°F); the differences in the 30 and 3000-second hardnesses characterizing the creep tendency of the alloy, are approximately the same at 500, 600 and 700°C.

4. Increase of the test temperature to 800 and 900°C (1470 and 1650°F) shows that the 25% molybdenum alloy (Ni-base) softens more readily than the alloy with 7% Mo.
5. At 1000°C (1830°F) nickel-base alloys suffer identical softening irrespective of their Mo contents, and the hardness/time curve is similar to that obtained at 500-600°C.
6. Increase of the Mo content within the range studied only slightly affects the elastic modulus, which continuously decreases with increase of test temperature.
7. The logarithmic decrement of elastic vibrations does not change up to 500°C (930°F) in any of the alloys studied, but with further increase of test temperature, there is a sharp increase in internal friction; this process starts in one-phase alloys at a lower temperature than in two-phase alloys.

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