CANADA

EFFECTS OF COLD WORK AND QUENCHING ON THE MAGNETIC SUSCEPTIBILITY OF A COMMERCIAL TITANIUM ALLOY

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REPRINTED FROM AMERICAN SOCIETY FOR
METALS, NO. 114; VOL. 51, TRANS. 1958

DEPARTMENT OF MINES AND
TECHNICAL SURVEYS, OTTAWA

MINES BRANCH
RESEARCH REPORT

R 35

PRICE 25 CENTS

1958

EFFECTS OF COLD WORK AND QUENCHING ON THE MAGNETIC SUSCEPTIBILITY OF A COMMERCIAL TITANIUM ALLOY

By Y. L. Yao

Abstract

The mean susceptibility of Ti-140A measured at room temperature increases with cold work and quenching. While the annealing of most cold-worked and all quenched samples, at 300 °C (570 °F) for 4 hours, almost wipes out this increase, the identical annealing of lightly deformed samples may cause a further increase. It is shown that the anomalous effects of low-temperature annealing may be connected to strain aging. Tentative explanations as to what causes the change of the mean susceptibility have been made and some applications are given.

THE EFFECTS OF cold work on the electrical resistance of a metal or on the magnetic properties of a ferromagnetic metal have been investigated by a number of workers both theoretically and experimentally (1,2).[1] In contrast to these, no adequate theory has been suggested to predict the effects of cold work on the magnetic susceptibility of a non-ferromagnetic metal and the experimental results of a few workers on cubic metals are contradictory (3).

In the case of non-cubic metals, the magnetic susceptibilities are, in general, anisotropic. A single determination of susceptibility of anisotropic polycrystalline metals in any arbitrary direction will yield little information. However, it has been shown (4) that the arithmetical mean of the susceptibilities measured in any three orthogonal directions is a constant for a single crystal or a polycrystalline sample. Thus, the mean susceptibility is independent of the degree of preferred orientation and any change in value of the mean susceptibility after cold work must be ascribed to other causes.

By previous experiments in this laboratory (5), it was found that the mean paramagnetic susceptibility of commercially pure titanium increases about 2% after heavy cold work and this increase is almost wiped out after annealing at 300 °C (570 °F) for 4 hours. Recent ex-

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[1] The figures appearing in parentheses pertain to the references appended to this paper.

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periments indicate that the similar increase of a commercial titanium alloy, Ti-140A, may be as high as 4%. It was decided that a detailed study of this alloy would be carried out. The investigation would cover the effects of cold work, quenching and annealing on the mean susceptibility.

EXPERIMENTAL METHOD

The Ti-140A used for this investigation has the following analysis in weight percentages: Fe, 2.29; Cr, 2.04; Mo, 1.74; Mn, 0.028; Cu, 0.011; W, 0.003; C, 0.043; 0, 0.11; N, 0.021; H, 0.0005; Ti, the remainder. According to the nomenclature system of titanium alloys proposed by Lippert (6), this alloy will be designated as T 96015.

Throughout this investigation, except for compression tests, the starting material was a ⅛ inch sheet.

Three kinds of cold work were used: rolling, extension, and compression.

For the rolling tests, strips of 2 inch x ½ inch were cut out from the ⅛-inch sheet, wrapped in a thin sheet of the same alloy and annealed in vacuum with a pressure less than 0.1 micron of mercury at 960 °C (1760 °F) for 4 hours followed by slow cooling. This particular treatment has been found from previous measurements to result in the polycrystalline alloy being magnetically isotropic to within ½% and in a uniform grain size of about 0.15 mm. Samples were cut out from a region near the center of the cross section of individual strips, after passing between small rollers to different degrees of reduction. The strips were always rolled in the same direction and at room temperature.

For the extension tests, small tensile specimens of 1-inch gage length, after homogenizing treatment, were stretched at room temperature in a Hounsfield tensometer. The alloy fractured at 8% elongation. The samples were cut from a region near the center of reduced cross section or near the fracture.

For the compression tests, the sheet was cut into suitable sizes, melted in an argon-filled furnace, cast into a small button, hot-swaged and hot-pressed into an approximately 1¼-inch cube. After the oxidized layer was ground off, the samples were cut out, homogenized and compressed between hardened-steel grips at room temperature in a compression testing machine.

For the quenching tests, identical small tensile specimens of 1-inch-gage length, after homogenizing treatment, were placed in different Vycor capsules, two in each. The capsules were evacuated, sealed and heated at between 300 and 1000 °C (570 and 1830 °F) for 4 hours. Then they were quenched in running water near room temperature. Samples were cut out from one specimen as quenched and from the other after being stretched at room temperature in the Hounsfield tensometer to produce a 2% elongation.

Three kinds of annealing were used: The rolled samples, the quenched samples, and the quenched and then stretched samples, were annealed at 300 °C (570 °F) for 4 hours. A set of three 50% compressed samples were individually annealed at 300, 400 and 500 °C (570, 750 and 930 °F). The total time of annealing at each temperature was 24 hours. The annealing was interrupted at different time intervals for magnetic measurements. The stretched samples were successively annealed from 300 to 800 °C (570 to 1470 °F), the time of annealing at each temperature being 4 hours.

Magnetic susceptibilities were measured by using the Faraday method. The experimental arrangement was described before (5). It is only necessary to repeat here that special pole-pieces were made (7) and fitted to the electromagnet. Such an arrangement gives a reasonable volume having a uniform value of H(dH/dy), which satisfies the experimental conditions under which three orthogonal susceptibilities may be determined for polycrystals of arbitrary preferred orientation (4). The weight of a sample was from 0.15 to 0.25 gram. The sample was always slightly etched in a diluted mixture of nitric and hydrofluoric acid and appropriately cleaned. All susceptibility measurements were made at a temperature of 25 ±2 °C. A Honda-Owen plot of apparent susceptibility against the reciprocal field was made for each measurement. All susceptibility values reported were taken as the arithmetical mean of three orthogonal susceptibilities. Duplicate measurements from equivalent samples could be reproduced to within 0.2%, although there may be a constant error of up to 1% in all of the measurements due to the error in measuring the magnetic field intensity.

EXPERIMENTAL RESULTS

The effects of cold work on the mean susceptibility are shown in Fig. 1. In each type of cold work the deformation curves show a more or less pronounced maximum. It is to be noted that for the same linear deformation, stretched samples are characterized by a much more rapid increase of $\bar{\chi}$.

The effects of quenching are shown in Fig. 2. It is seen that the $\bar{\chi}$ increases steadily with the rise of quenching temperature up to 1000 °C (1830 °F) and that the extension following the quenching results in a decrease of $\bar{\chi}$.

After rolled samples, quenched samples, and quenched and then stretched samples were annealed at 300 °C (570 °F) for 4 hours, the $\bar{\chi}$ had all returned to 0.3% of the original value.

Compressed samples behaved somewhat differently and in the 50% compression case the $\bar{\chi}$ had not returned to its original value even after a 24 hour anneal at 300 °C (570 °F). At higher temperature the recovery was more complete. This behavior is shown in Fig. 3.

The results of successive annealing of stretched samples are shown in Fig. 4. For the slightly stretched samples the susceptibility increases

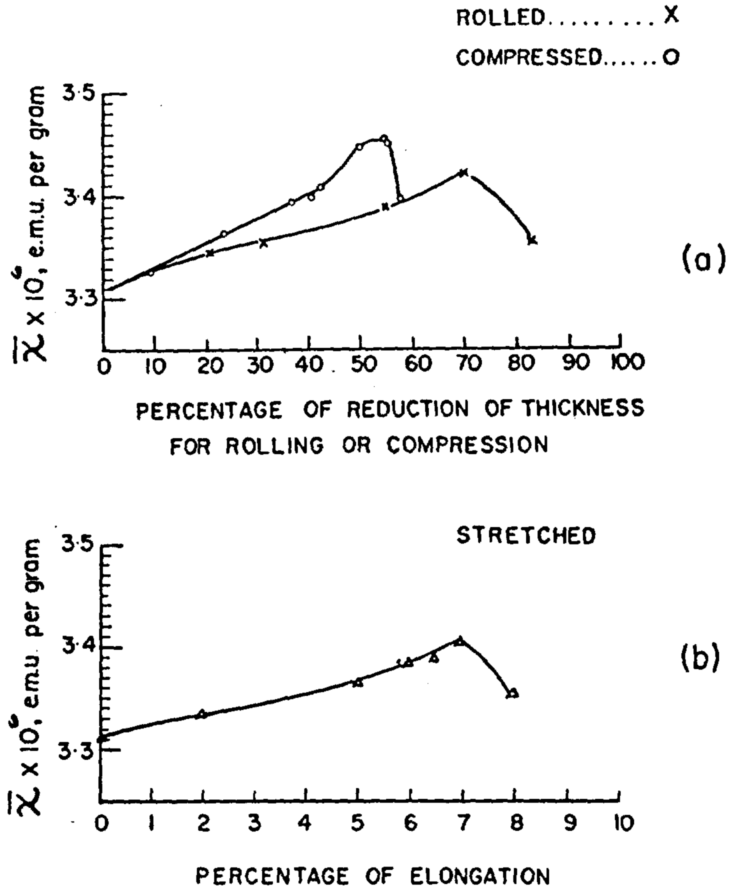

Fig. 1—Deformation Curves: Mean Magnetic Susceptibility Versus Degree of Cold Work. (a) Rolling and compression. (b) Extension.

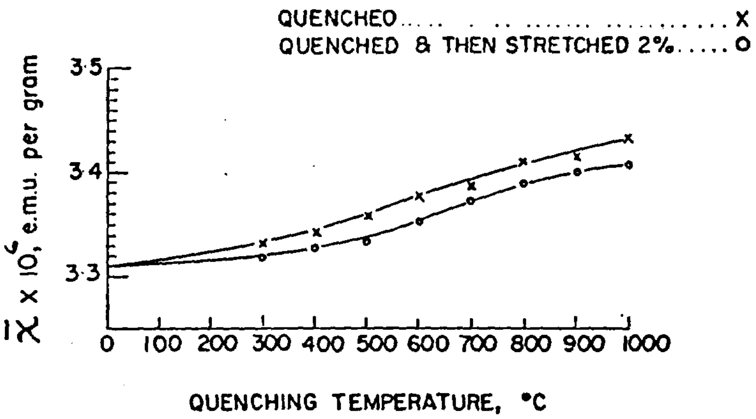

Fig. 2—Quenching Curves: Mean Magnetic Susceptibility Versus Quenching Temperature.

to a peak value after the 400 °C (750 °F) anneal; while for the larger extensions no such increase was detected, and recovery is practically complete in the temperature range of 400 and 500 °C (750 and 930 °F). All samples show another weaker peak after annealing in the temperature range of 600 and 700 °C (1110 and 1290 °F), but the original value is almost restored after annealing at 800 °C (1470 °F).

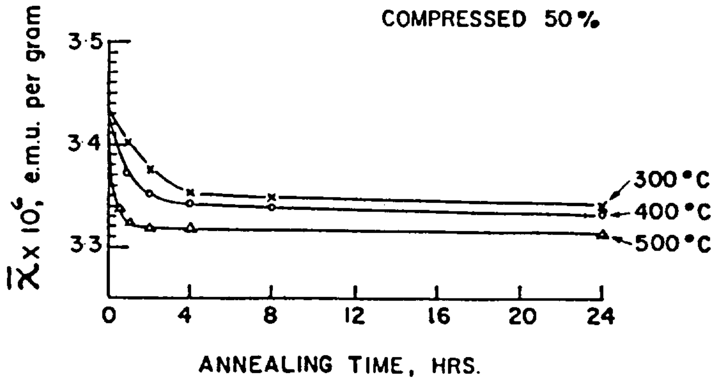

Fig. 3—Isothermal Annealing Curves: 50% Compressed Samples Annealed at 300, 400 and 500 °C (570, 750 and 930 °F).

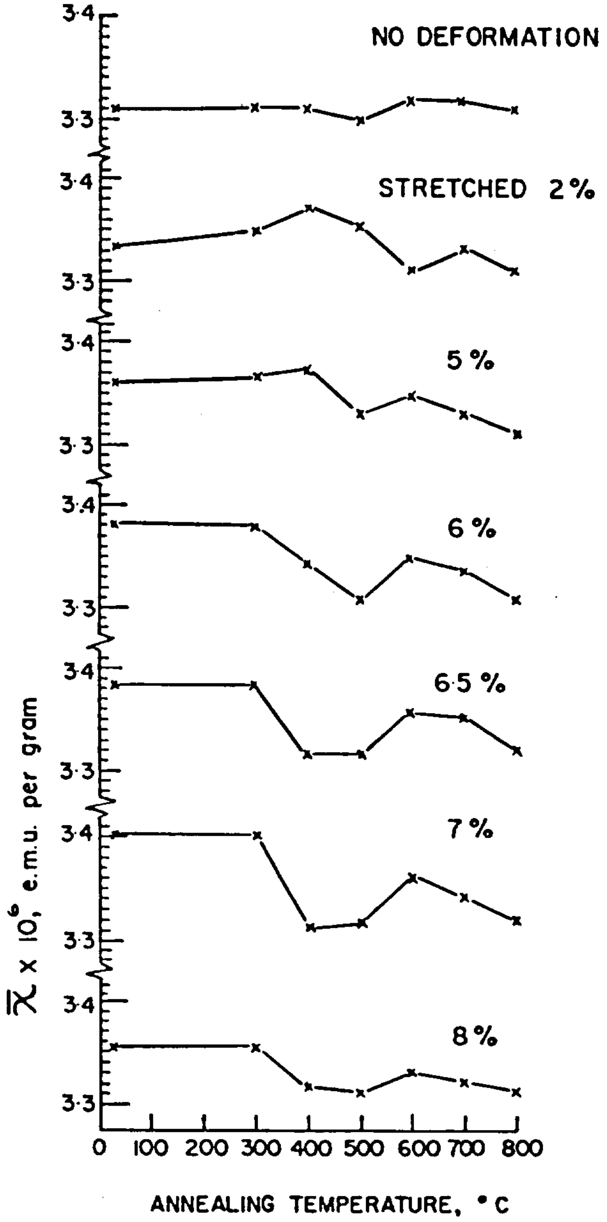

Fig. 4—Isothermal Annealing Curves: Stretched Samples Annealed Successively from 300 to 800 °C (570 to 1470 °F), 4 Hours at Each Temperature.

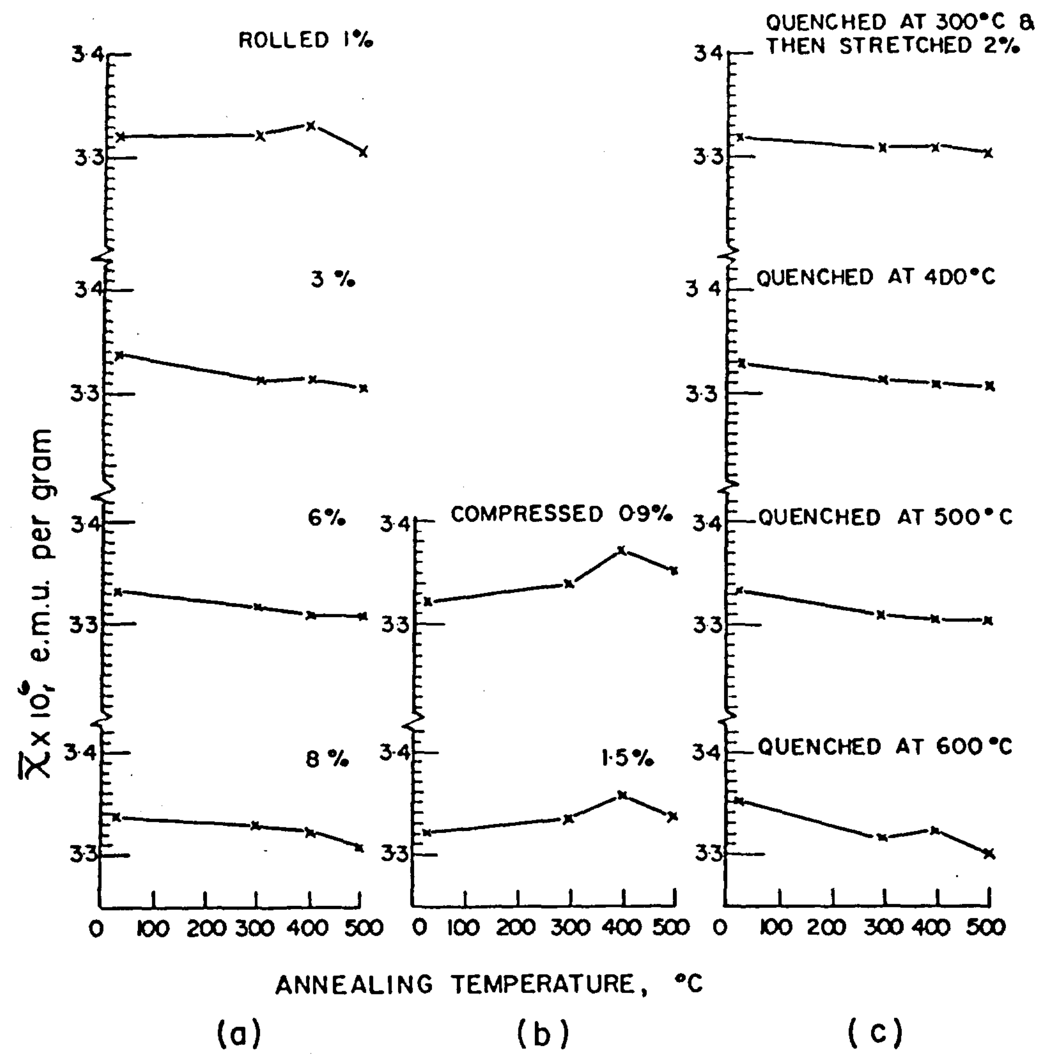

Fig. 5—Isochronal Annealing Curves: Samples Annealed Successively from 300 to 500 °C (570 to 930 °F), 4 Hours at Each Temperature. (a) Lightly rolled. (b) Lightly compressed. (c) Quenched and then stretched 2%.

To ascertain whether the peak attained after annealing at 400 ° C (750 °F) is confined to stretching, some lightly rolled and compressed samples, and some samples quenched from below 700 °C (1290 °F) and then stretched 2% were isochronally annealed. The results are shown in Fig. 5. It is seen that samples rolled 1% or compressed 0.9 and 1.5% all show a slight increase but quenched and then stretched samples do not.

DISCUSSION

The factors which might change $\bar{\chi}$ are changes in ferromagnetic iron, interstitial impurities, hardness and internal strains. As to the contamination or precipitation of ferromagnetic iron, the Honda-Owen plot was used to estimate the amount of ferromagnetic iron. In spite of the presence of about 2% of total iron, the amount of ferromagnetic iron varies from 5 to 10 parts per million by weight. The variation is random even for samples quenched from different temperatures. Furthermore, except for lightly deformed samples, the increase of $\bar{\chi}$ may be almost wiped out by annealing at 300 °C (570 °F) for 4 hours. It is unlikely that any ferromagnetic iron would redissolve at this low temperature. Because the initial homogenizing and all other heat treatments were performed in vacuum, the hydrogen content would remain low and there was no contamination of other interstitial impurities. Although there is superficial correlation between hardness and $\bar{\chi}$, the annealing out of the $\Delta\bar{\chi}$ at 300 °C (570 °F), which is well below the recrystallization temperature, will rule out any direct connection.

One reasonable assumption is that the inducement or relief of elastic strains will change the value of $\bar{\chi}$. Thus the falling of $\bar{\chi}$ after a certain degree of cold work (Fig. 1) may be due to the partial relief of stresses caused by microscopic fissures. Cracks were observed on the edges of heavily rolled strips and on heavily compressed samples. It was also observed that stretching after quenching decreases the $\bar{\chi}$ (Fig. 2). The explanation of this may be that stretching reduces quenching stresses (8).

The wiping out of $\Delta\bar{\chi}$ after low-temperature annealing is due to the familiar stress-relief treatment. Indeed, the isothermal annealing of 50% compressed sample at 300, 400 and 500 °C (570, 750 and 930 °F) (Fig. 3) gives some idea about the rate of relaxation of internal stresses. If we calculate the efficiency of stress relief from one minus the ratio of the instantaneous $\Delta\bar{\chi}$ to the original $\Delta\bar{\chi}$, internal stresses were 81% relieved in 1 hour at 500 °C (930 °F), 56% at 400 °C (750 °F) and 33% at 300 °C (570 °F). These figures may be compared with those of Bernstein (9) on rolled samples of Ti-150A. He used a spring-back method to measure the efficiency of stress relief. His figures are as follows: 92% relieved in 1 hour at 800 °F (427 °C), 47% at 600 °F (316 °C) and 22% at 400 °F (205 °C).

Although the reasons for the development of a weak peak at the temperature range of 600 and 700 °C (1110 and 1290 °F) for stretched samples (Fig. 4) are not known (this being also observed in a fresh sample), the slight increase of $\bar{\chi}$ after the 300 °C (570 °F) anneal and the peak developed after the 400 °C (750 °F) anneal appear to be associated with strain aging. Strain aging could lead to a suppression of low temperature recovery (10). Titanium (11) and some titanium alloys such as Ti-150A and RC-130B (12) are susceptible to strain aging. To test whether Ti-140A belongs to this category, tensile specimens of 3-inch-gage length were homogenized, stretched to a plastic strain of 2% at room temperature, heated in vacuum from 300 to 800 °C (570 to 1470 °F) for 1 hour and then retested in tension at room temperature. The results are shown in Fig. 6. The mild tendency to form a yield point after annealing at 300 or 400 °C (570 or 750 °F) and the strengthening of the room temperature curve after annealing at

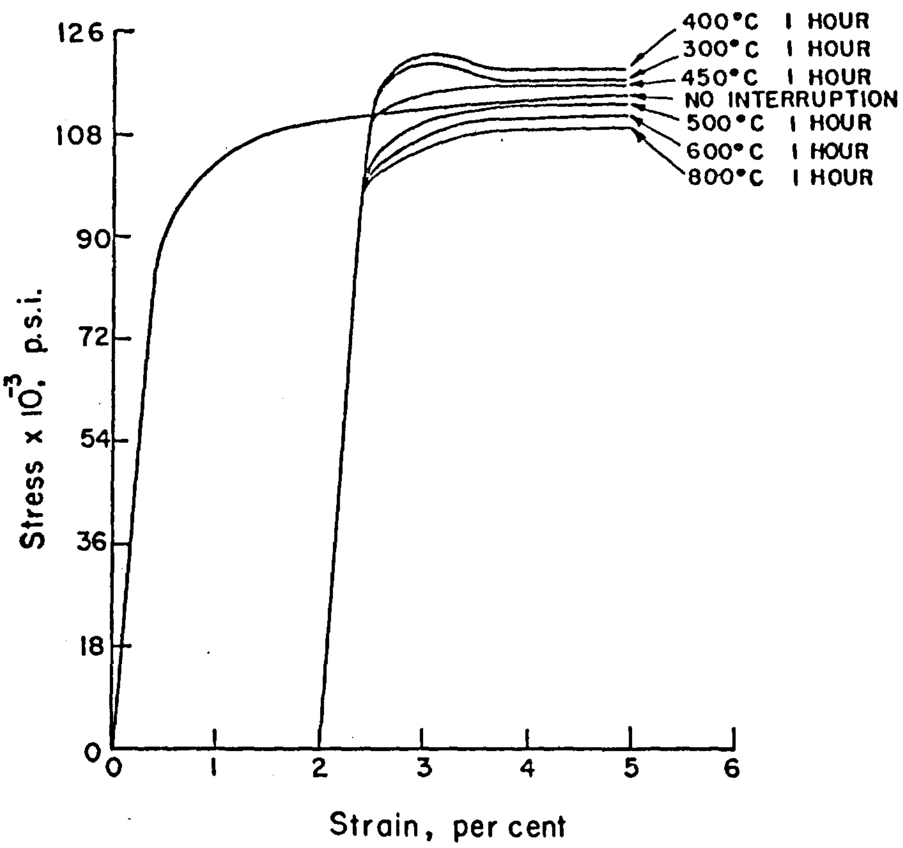

Fig. 6—Effects of Aging Temperature on the Stress-Strain Curve at Room Temperature.

300, 400 or 450 °C (570, 750 or 840 °F) indicate that strain aging effects are involved. The maximum temperature of strain aging is about 400 °C (750 °F). So far we have shown that strain aging and the increase in $\bar{\chi}$ occur in the same temperature range and that the increase in $\bar{\chi}$ is suppressed by overstrain. It is known that strain aging is suppressed by overstrain (for example, Table II of Rostoker and Yamamoto's article (13) illustrates this for vanadium). Hence we are tempted to conclude that these two are related.

Based on thermodynamical principles, it is known that the partial differential coefficient of intensity of magnetization with respect to stress is equal to that of strain with respect to magnetic field. If the assumption that internal strains increase the intensity of magnetization is correct, we shall expect that the magnetostriction of this alloy is not too small to be measured.

CONCLUSIONS

a. The mean paramagnetic susceptibility of solid polycrystals of Ti-140A at room temperature is $3.31_1 \times 10^{-6}$ e.m.u. per gram.

b. Cold work and quenching at high temperature up to 1000 °C (1830 °F) increase the mean susceptibility measured at room temperature. The maximum increase is about 4%.

c. Except for lightly deformed samples, in most cases the increase is almost wiped out by annealing at 300 °C (570 °F) for 4 hours.

d. For lightly deformed samples, annealing at 300 °C (570 °F) for 4 hours may bring out an increase in the mean susceptibility and a further annealing at 400 °C (750 °F) for 4 hours may develop a peak.

e. The anomalous effects of low temperature annealing may be connected to strain aging.

f. A tentative assumption is that the change of the mean susceptibility is due to the inducement or relief of internal strains.

g. When the alloy is in the cold-worked or quenched state, the measurement of the mean susceptibility offers a quick means of detecting intense stress concentrations. If such a sample is annealed, the same measurement indicates the efficiency of stress relief. Also, for a lightly deformed sample the same measurement after annealing will indicate whether it is susceptible to strain aging.

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