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ANISOTROPIC DILATATION DURING ANNEALING OF 18% NICKEL MARAGING STEEL

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ABSTRACT

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The dimensional changes which occurred during annealing and aging of 18% nickel maraging steel sheet and plate were measured to determine the dimensional stability of this alloy. Significant anisotropic dimensional changes were measured during annealing, but only small isotropic shrinkage was observed during aging. Multiple annealing treatments revealed that the anisotropic dimensional changes occuring during annealing were repetitive and cumulative.

Recognition of the anisotropic dimensional changes during annealing is necessary since they are of sufficient magnitude to preclude any reannealing of precision or dimensionally critical components that are fabricated from 18% nickel maraging steel.

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ANISOTROPIC DILATATION DURING ANNEALING OF 18% NICKEL MARAGING STEEL

SUMMARY

The dimensional changes which occurred during annealing and aging of 18% nickel maraging steel sheet and plate were measured to determine the dimensional stability of this alloy. Anisotropic dimensional changes during annealing were measured of the order of -0.3% in the longitudinal and transverse directions and + 0.6% in the short transverse direction. During aging, only small isotropic dimensional changes of about - 0.05% were observed. Multiple annealing treatments revealed that the anisotropic dimensional changes occurring during annealing were repetitive and cumulative.

INTRODUCTION

In the process of heat treating 18% nickel maraging steel plate, anisotropic dimensional changes were noted after annealing. Since these dimensional changes appeared to contradict published information (Ref. 1) regarding the dimensional stability of maraging steels, an investigation was undertaken to characterize the observed changes. Recognition of these anisotropic dimensional changes during annealing is necessary since they are of sufficient magnitude to preclude any reannealing of precision or dimensionally critical components that are fabricated from 18% nickel maraging steel.

One of the advantages of the 18% nickel maraging steel over other high strength alloys is the simplicity of heat treatment procedures. The 18% nickel maraging steel is a low carbon, iron-nickel alloy containing cobalt and molybdenum. At room temperature, this alloy has a martensitic structure which is body-centered cubic and is ductile and formable. Only two steps, annealing and aging, are involved in heat treating this alloy. Generally, this material is fabricated in the annealed condition and then aged to attain high strength.

The annealing process consists of one hour at 816°C (1500°F) and air cooling to room temperature. During heating to the 816°C (1500°F) annealing temperature, the martensite is transformed to austenite. Upon continuous heating, this transformation begins at about 590°C (1100°F) and is complete at approximately 770°C (1420°F). Upon cooling, the structure begins to transform from austenite to martensite at about 154°C (310°F) (M_s temperature). This transformation is reported to be complete at about 99°C (210°F) (M_f temperature) (Ref. 2). However, the results of the present investigation indicated a somewhat lower M_f temperature.

The martensitic transformation in iron-nickel alloys is essentially independent of cooling rate. Jones et al. have found that the austenite-martensite transformation is independent of heating or cooling rate in the range of 2°C to 150°C/min (3.6°F to 270°F/min) (Ref. 3). Insensitivity of the martensitic transformation to cooling rate eliminates section size effects and quenching problems and provides the 18% nickel maraging steel with a distinct advantage over other steels.

The annealing cycle, thus, includes a martensite to austenite to martensite sequence of transformations. Although a dimensional change is anticipated on transforming from one phase to the other, a net dimensional change is not necessarily indicated for this sequence of transformations.

The aging treatment consists of heating for three hours at 482°C (900°F) and air cooling to room temperature. No transformations occur during this treatment. The aging mechanism responsible for strengthening the 18% nickel maraging steel has not been established definitely; however, some evidence for ordering (Ref. 4) and precipitation of Ni_3Mo (Ref. 2) has been reported. Only small dimensional changes are observed during aging (Ref. 1).

This investigation was concerned primarily with the measurement of the anisotropic dimensional changes accompanying annealing. Published reports have stated repeatedly that the 18% nickel maraging steel is relatively free of dimensional changes during heat treatment. Although this is true for the aging treatment, apparently it is not true for the annealing treatment. Somehow, this fact has been overlooked or avoided by other investigators. Also, as far as can be determined, no report has been made of the anisotropy in the dimensional changes as observed in this investigation.

EXPERIMENTAL PROCEDURES

The 18% nickel maraging steel used in this study was produced by Republic Steel Corporation. Basic data for this material are given in Table I. Two different sample geometries were used for dimensional studies. Dilatometer specimens, approximately 0.1 x 0.1 x 1.96 inches, were cut from 0.1-inch thick sheet and 0.5-inch thick plate stock. Block specimens, 0.5 x 1.0 x 1.5 inches, were machined from the 0.5-inch plate. Dilatometer specimens were taken from the longitudinal and transverse directions. Short transverse direction dilatometer specimens were made up of four sections which had been cut from the short transverse direction (plate thickness) of the 0.5-inch thick plate.

All heat treating was conducted in vacuum to minimize oxidation of the specimens. Dilatometric measurements were made with a Leitz dilatometer equipped with a vacuum attachment. Vacuum achieved in this system was approximately 10^{-3} torr. Block specimens were heat treated in a vacuum furnace at approximately 10^{-5} torr. Dimensional changes of the block specimen were measured with a micrometer to 0.0001 inch.

Metallographic studies were made of selected specimens to check for structural changes after various heat treatments. Rockwell hardness measurements were made to check mechanical properties after heat treating.

RESULTS

Two types of experiments were conducted to study the dimensional changes associated with the heat treatment of 18% nickel maraging steel. In the first type, the dimensional changes accompanying a single annealing and aging treatment were evaluated. In the second set of experiments, the effect of multiple anneals was investigated. The purpose of these latter tests was to gain a better understanding of the mechanism responsible for the dimensional changes accompanying annealing and to demonstrate more clearly the anisotropic nature of these changes.

Single Cycle Annealing

Simple measurements of both the dilatometer and block specimens of 18% nickel maraging steel revealed that dimensional changes of significant magnitude accompany annealing and that these changes are anisotropic. The maraging steel specimens contracted in the longitudinal and transverse directions and expanded in the short transverse (plate thickness) direction. The magnitude of the dimensional changes observed in the 0.1-inch thick sheet and 0.5-inch thick plate is given in Table II. The anisotropy observed would not be expected normally in a cubic material.

Dilatometer curves obtained for longitudinal, transverse, and short transverse directions are shown in FIG 1. These curves indicate the changes in length of the dilatometer specimens as a function of temperature. Each curve shows the relatively uniform rate of expansion experienced upon heating the martensitic structure from room temperature to about 560°C (1040°F). Above this temperature, the dimensional change is erratic because of the simultaneous contraction associated with the transformation of martensite to austenite and the expansion of the residual martensite. At approximately 770°C (1420°F), the transformation is complete with the structure 100% austenite. Upon cooling, a relatively uniform contraction of the austenitic phase is experienced until approximately 154°C (310°F), where a rapid expansion occurs because of the transformation of austenite to martensite. This transformation is completed at about 60°C (140°F). Note that in all cases a net change in dimensions is recorded after annealing. The change is negative for the longitudinal and transverse directions and positive for the short transverse direction.

Two major features of the dilatometer curves should be noted. First, the dilatometer curves indicate that the dimensional change measured upon heating the martensitic structure and cooling the austenitic structure is isotropic, i.e., the slopes of the curves for longitudinal, transverse, and short transverse, with the exception of the transformation regions, are essentially equal. This isotropy is characteristic of a cubic material. The second feature, which indicates the source of the dimensional anisotropy, is the difference in contraction and expansion characteristics during the phase transformations. This difference is particularly evident in comparing the dilatometer curves obtained in the short transverse direction with those obtained in the longitudinal and transverse directions. It is apparent that the anisotropy, measured after annealing, must be associated with the differences in dimensional changes observed during the martensite to austenite and austenite to martensite phase transformations.

To establish that the martensitic transformation was complete above room temperature, a dilatometer curve was obtained for a longitudinal sample between -196°C (-320°F) and 816°C (1500°F). This curve, shown in FIG 2, demonstrated that no further transformation occurs below room temperature. Therefore, the net change in dimensions cannot be attributed to incomplete transformation at room temperature.

Aging

The dimensional changes accompanying aging were small and isotropic. A net shrinkage of about 0.05% was measured. Such shrinkage is common during aging and is attributed to precipitation of alloying elements from solid solution. Dilatometer curves for a standard three-hour age at 482°C (900°F) are shown in FIG 3. Only the curves for the longitudinal and short transverse directions are shown since the curve for the transverse direction is similar to the longitudinal direction.

Multiple Cycle Annealing

Although, in practice, multiple annealing is not likely to occur, a series of dilatometer and block specimens were annealed a number of times to gain additional information on the anisotropic dimensional changes in 18% nickel maraging steel. These tests showed that the anisotropic changes were repetitive and cumulative. This is demonstrated by the dilatometer curves for multiple annealed transverse and short transverse direction specimens, shown in FIG 4 and 5. The transverse curves are typical of the curves obtained in the longitudinal direction also.

Each successive annealing treatment resulted in a net dimensional change, i.e., shrinkage in the transverse and longitudinal directions, and expansion in the short transverse direction. The cumulative effect is also demonstrated by the photographs in FIG 6. Figure 6a illustrates the dimensional changes introduced in a dilatometer specimen cycled to 816°C (1500°F) 35 times,and FIG 6b illustrates the dimensional changes occurring in a block specimen cycled to the annealing temperature 25 times. The anisotropy of the dimensional changes is readily apparent. It should be noted that it was not necessary to hold the specimen at the annealing temperature for any set time. All that was required to affect the dimensional change was the martensite to austenite to martensite sequence of transformations.

Figure 7 illustrates the progress of the dimensional changes in a block specimen cycled 25 times. A gradual decrease in the magnitude of the dimensional changes is evident as the number of cycles increases. Also included on this graph is a record of the hardness measured after each cycle. These hardness values are remarkably consistent; multiple annealing does not grossly affect the mechanical properties of this maraging steel. This observation is consistent with the metallographic observations. Figure 8 illustrates the microstructure of the 18% nickel maraging steel after 1 annealing cycle and after 35 annealing cycles. The microstructures are similar. Some coarsening of the structure appears to be evident after 35 cycles, but the evidence is not conclusive. A coarsening of the martensitic structure would result from a coarsening of the austenitic structure during annealing. Such a coarsening is possible since it is known that excessive time in the austenitic range will cause an increase in the austenite grain size (Ref. 2). It is also recognized that in maraging steel the grain size is unaffected by transformation (Ref. 2). Therefore, a coarsening of the martensitic structure of cycled specimens could reflect a cumulative change in the austenitic grain size.

DISCUSSION

The pronounced anisotropy and cumulative nature of the dimensional changes, accompanying annealing of 18% nickel maraging steel, would appear to contradict existing published information. It is reported that these steels are relatively free of dimensional changes during and after heat treatment (Ref. 1). However, close examination of published data revealed that other investigators have encountered dimensional changes during heat treatment of iron-nickel alloys but have avoided or overlooked mention of them. For example, examination of the dilatometric data reported by Decker et al. (Ref. 1) indicates a net decrease in the length of the dilatometer sample after annealing. The magnitude of the change is about one-half that observed in this study. Also, in an early study of 15% nickel steel, Jones and Pumphrey (Ref. 3) noted that "the dilatation, in general, was not found to be reproducible to a high degree of accuracy." Thus, dimensional effects have been observed but not discussed in the past. No mention of the anisotropic dimensional changes has been found.

Formation of a new crystal lattice during the martensitic transformation requires atomic realignments which produce shape deformation. The shape deformationassociated with the formation of the martensite plate is believed to consist of both shear and tensile or compressive strain (Ref. 5). However, at present, it is unlikely that existing theories can explain or account for the dimensional changes and anisotropy measured in this investigation.

In addition to these shape strains, evidence of plastic deformation in the untransformed austenite has been reported (Ref. 6). Pronounced slip lines are evident in the austenite phase of partially transformed iron-nickel alloys. These slip lines are retained in the martensite plates upon completion of the transformation. Similar plastic strains have been observed in the newly-formed austenite during the transformation of martensite to austenite. The cumulative changes in dimension accompanying annealing of the 18% nickel maraging steel are probably the result of this plastic deformation of the austenite matrix. Since it is unlikely that this plastic deformation is reversible, the deformation resulting from each successive transformation could accumulate.

Although evidence of plastic deformation during the martensite to austenite transformation exists, it does not, in itself, explain the anisotropy observed in the present investigation. The anisotropy may be traceable to the influence of a preferred orientation in the rolled plate. In the presence of a strong orientation, plastic deformation of the austenite during transformation reactions would be limited to specific directions since slip planes and directions would be oriented preferentially. Therefore, the magnitude and direction of the plastic deformation within the austenite might not be isotropic.

The gradual decrease in the relative magnitude of dimensional changes after many annealing cycles is consistent with an orientation produced anisotropy. Repetitive cycling could destroy the preferred orientation of the steel. This would create a more random structure in which the net dimensional changes in each direction--longitudinal, transverse, and short transverse--should diminish. Randomness of the structure should lead to randomness in the plastic deformation and a decrease in the magnitude of the net dimensional changes.

The preceding discussion, of course, is speculative since a thorough X-ray analysis of the 18% nickel maraging steel has not been made. Exploratory X-ray studies indicate that a rather extensive program will be required to develop a more clearly defined explanation of the dimensional anisotropy in this steel.

CONCLUSIONS

The basic results of this investigation of the dimensional stability of 18% nickel maraging steel demonstrated the following:

(1) Anisotropic dimensional changes accompany annealing with a contraction of about 0.3% in the longitudinal and transverse directions and an expansion of about 0.6% in the short transverse direction (plate thickness). The magnitude of these dimensional changes is sufficient to preclude reannealing of dimensionally critical components that are fabricated from 18% nickel maraging steel.

(2) These dimensional effects are repetitive and cumulative.

(3) The net changes in dimension cannot be attributed to incomplete transformation at room temperature.

(4) The magnitude of the dimensional changes diminishes slowly as the number of annealing cycles is increased.

(5) The only requirement for the anisotropic dimensional change is the martensite to austenite to martensite sequence of transformations.

(6) Aging results in a small isotropic shrinkage of about 0.05%.

TABLE I

Maraging Steel Data Sheet

Producer: Republic Steel Corporation

Material: RMS 300 MR Steel (Consumable Electrode Vacuum Melted)

*Composition %:

Nickel	18.80	Carbon	0.027
Cobalt	8.98	Manganese	0.05
Molybdenum	4.95	Phosphorus	0.006
Titanium	0.63	Sulfur	0.009
Aluminum	0.05	Silicon	0.01

*Typical Physical Properties: [Aged 3 hr at 482°C (900°F)]

	0.1" Sheet	0.5" Plate
Yield Strength (psi)	283,020	277,750
Tensile Strength (psi)	289,680	282,800
Elongation in 2" (%)	5	9
Hardness (R_c)	53	50

*Data supplied by Republic Steel Corporation

TABLE II

Dimensional Changes Accompanying Annealing
of 18% Nickel Maraging Steel
(One Annealing Treatment)

Specimen	Dimensional Change, %		
	Longitudinal	Transverse	Short Transverse
Dilatometer (0.1" x 0.1" x 1.96")	- 0.28	- 0.28	+0.60
Block (0.5" x 1.0" x 1.5")	- 0.36	- 0.31	+0.62

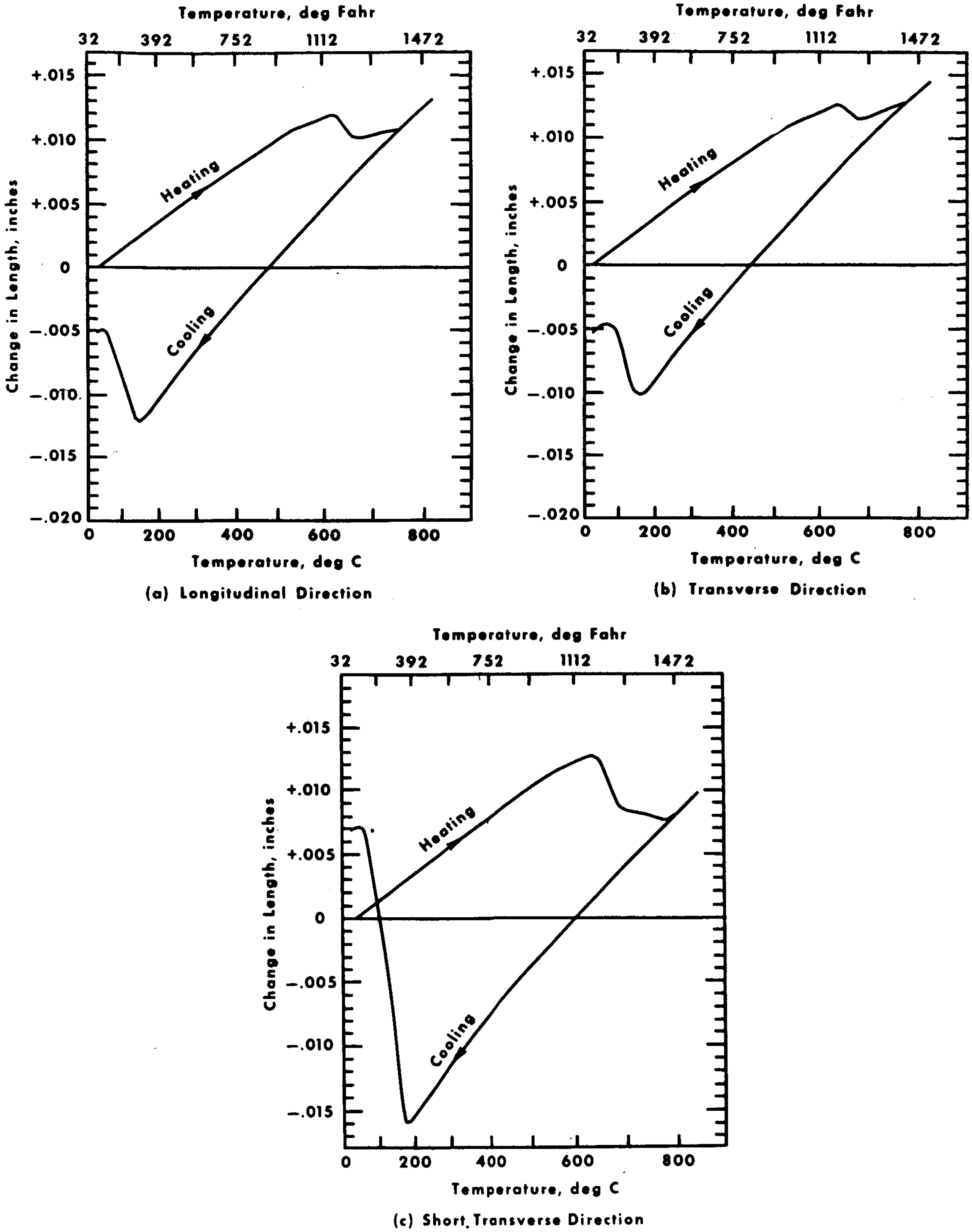


FIGURE 1 DILATOMETER CURVES FOR 18% NICKEL MARAGING STEEL DURING ANNEALING

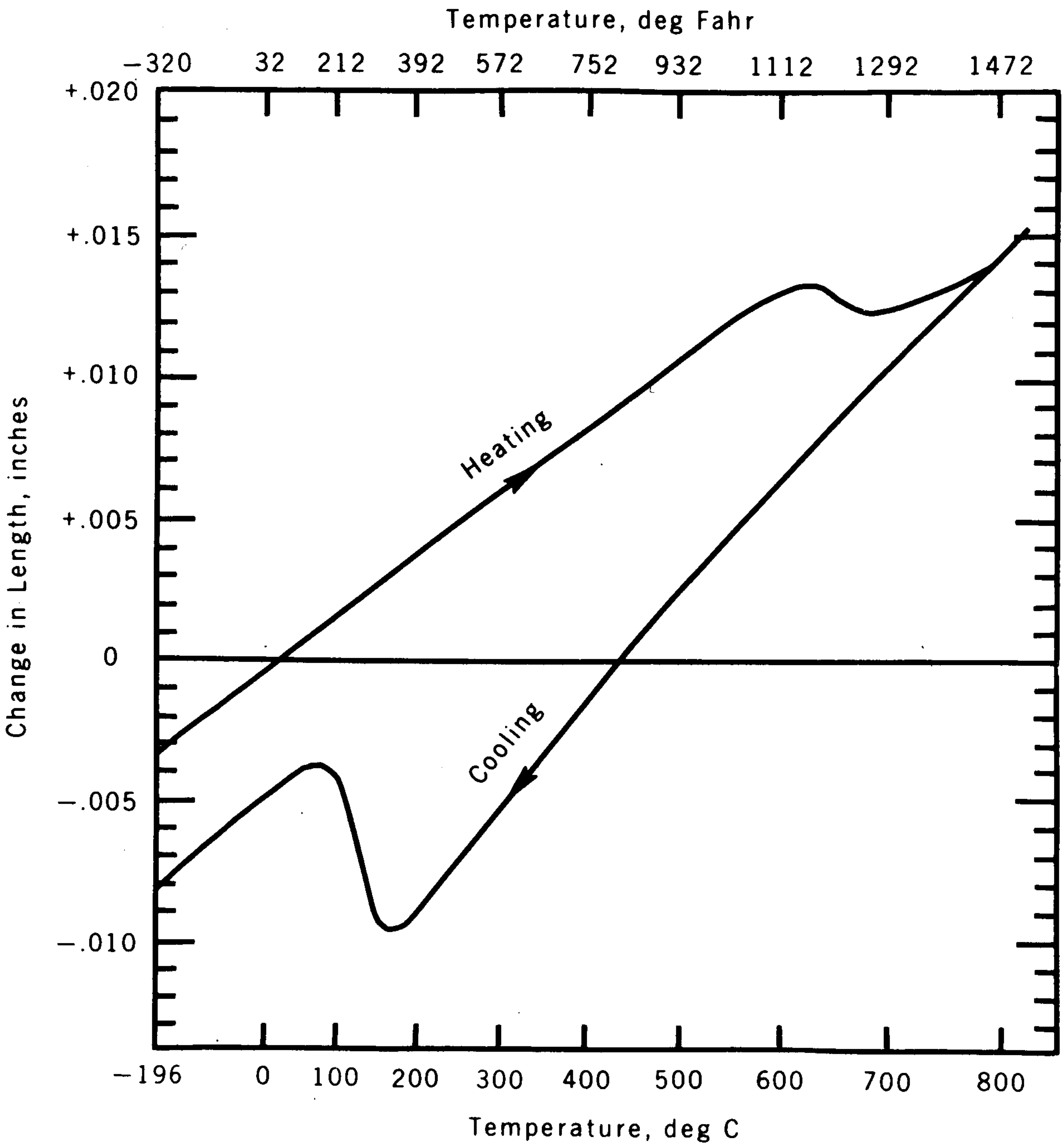


FIGURE 2 DILATOMETER CURVE FOR LONGITUDINAL DIRECTION OF 18% NICKEL MARAGING STEEL FROM −196°C TO ANNEALING TEMPERATURE

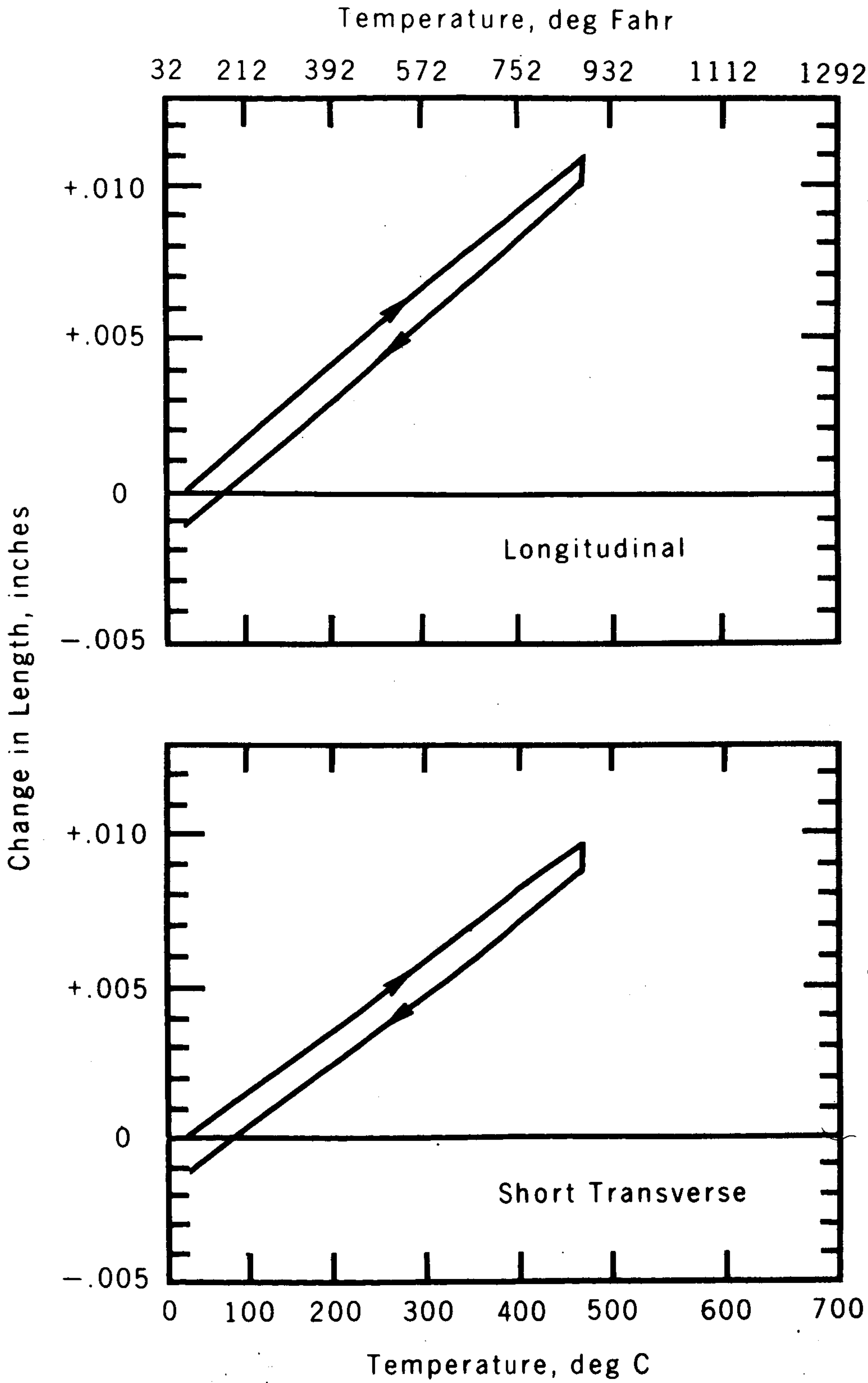


FIGURE 3 DILATOMETER CURVES FOR 18% NICKEL MARAGING STEEL DURING AGING CYCLE

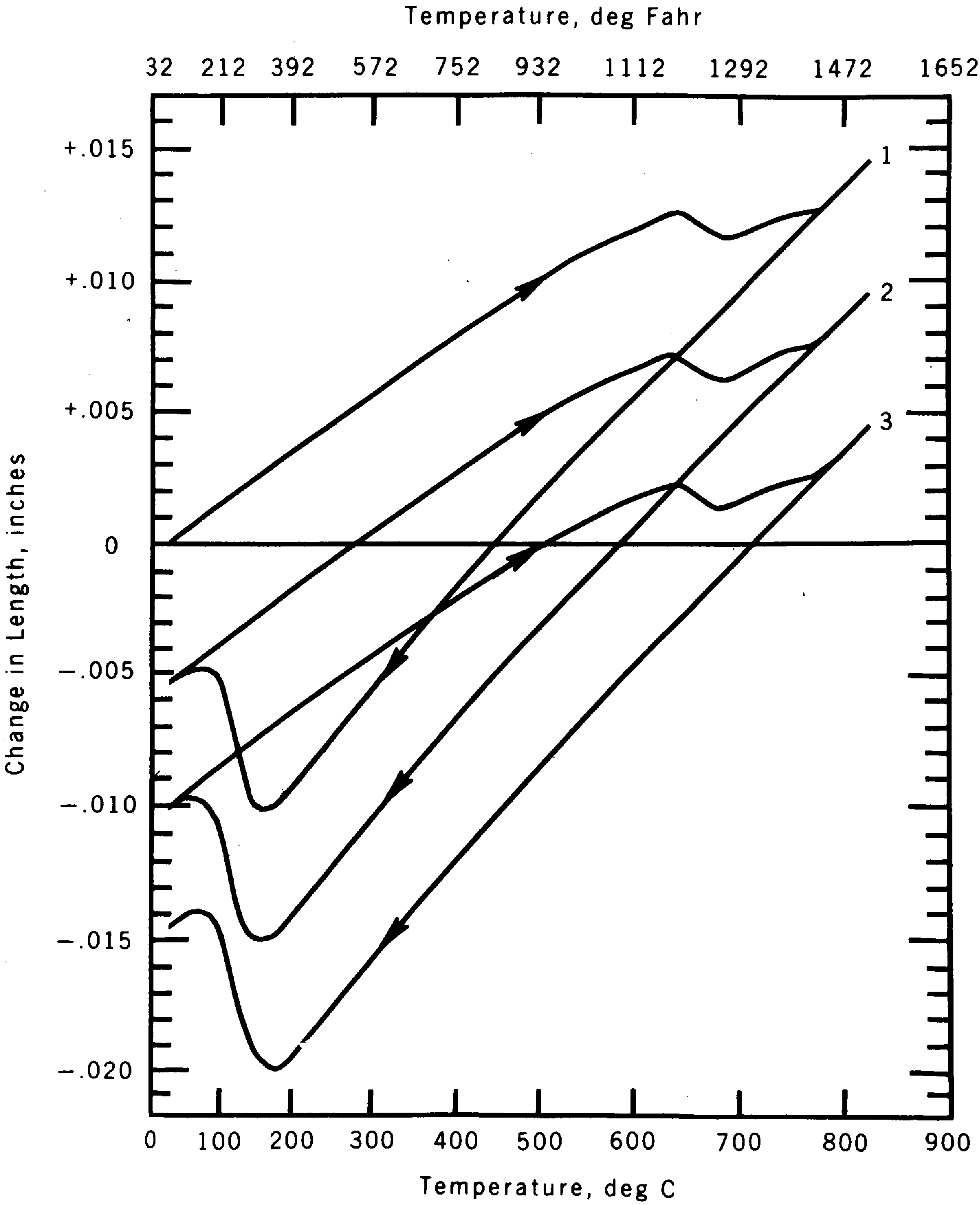


FIGURE 4 DILATOMETER CURVES FOR THREE SUCCESSIVE ANNEALS OF 18% NICKEL MARAGING STEEL (TRANSVERSE DIRECTION)

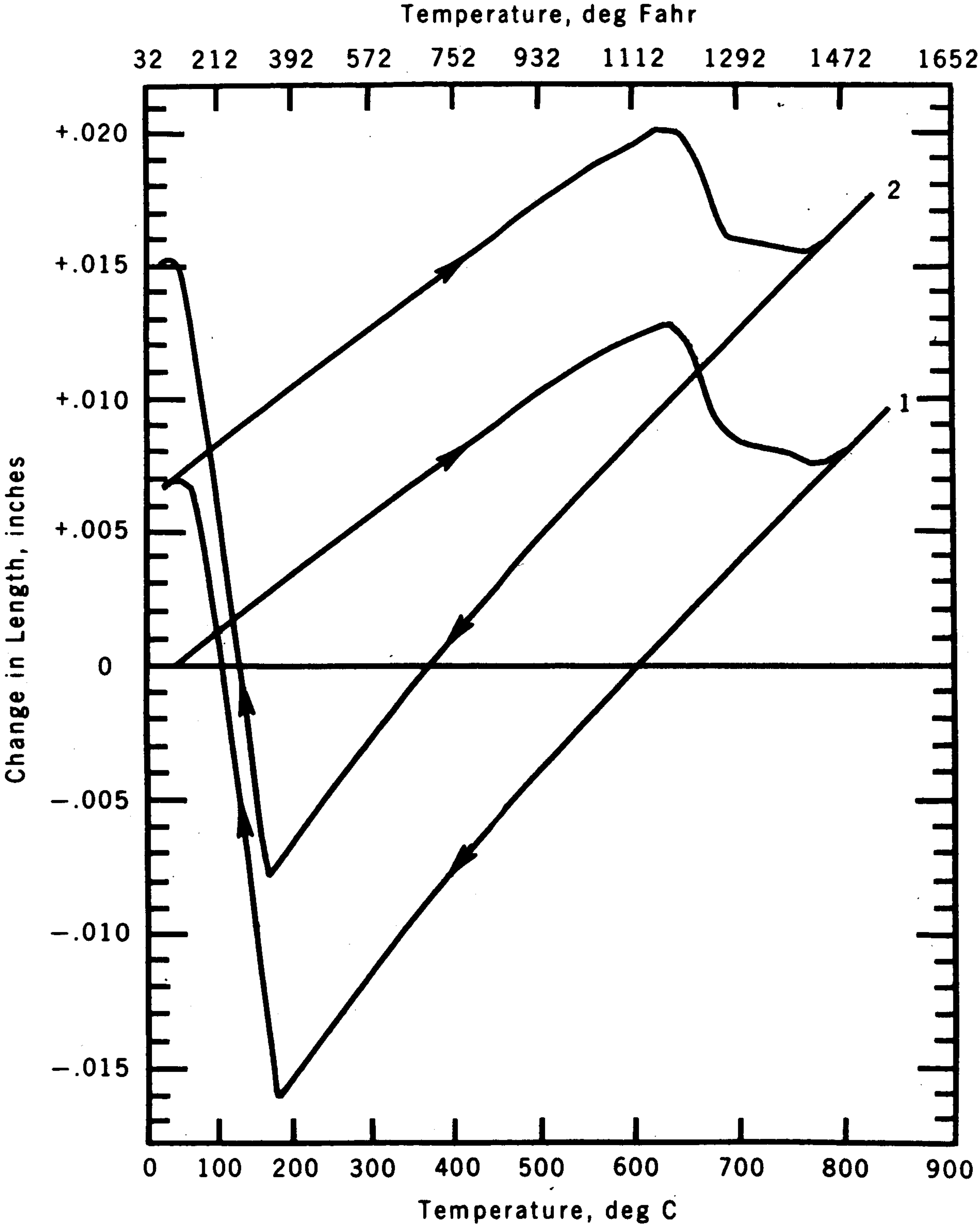
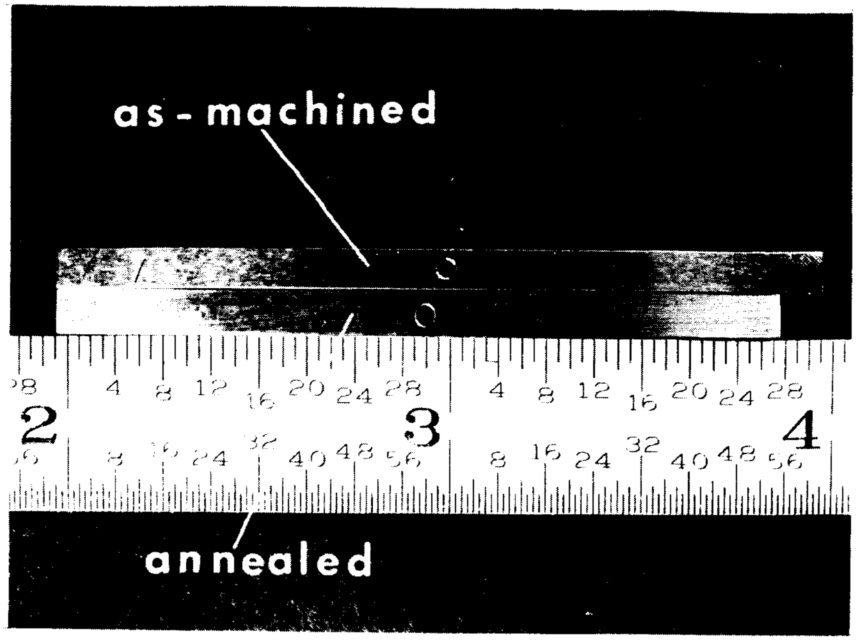
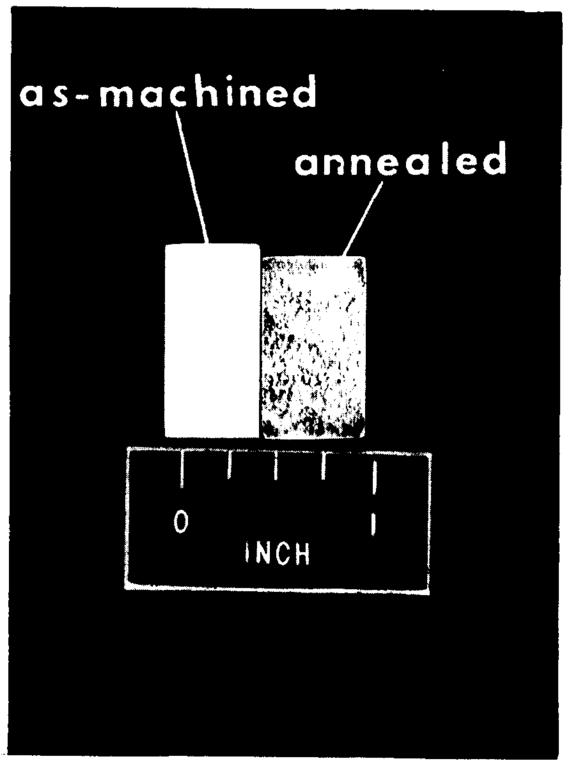


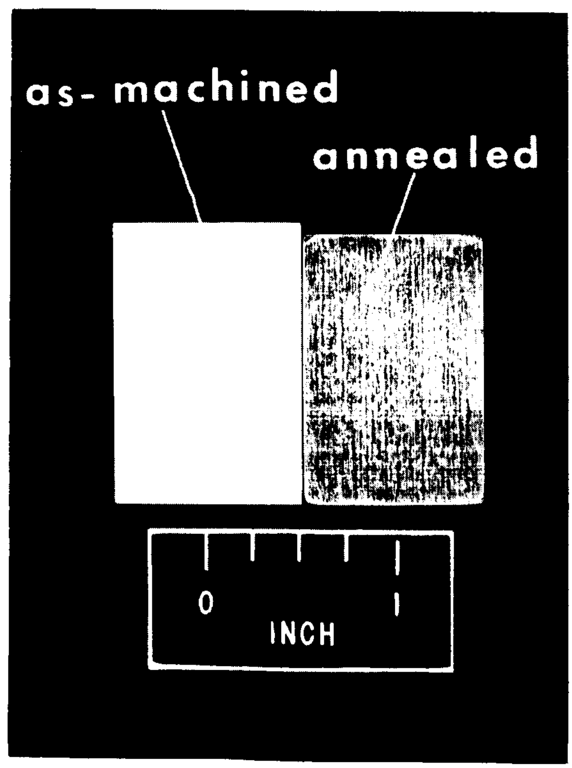
FIGURE 5 DILATOMETER CURVES FOR TWO SUCCESSIVE ANNEALS OF 18% NICKEL MARAGING STEEL (SHORT TRANSVERSE DIRECTION)



Dilatometer Specimen, 35 Anneals



Top View



End View

Block Specimen, 25 Anneals

FIGURE 6 COMPARISON OF AS-MACHINED AND MULTIPLE ANNEALED SAMPLES

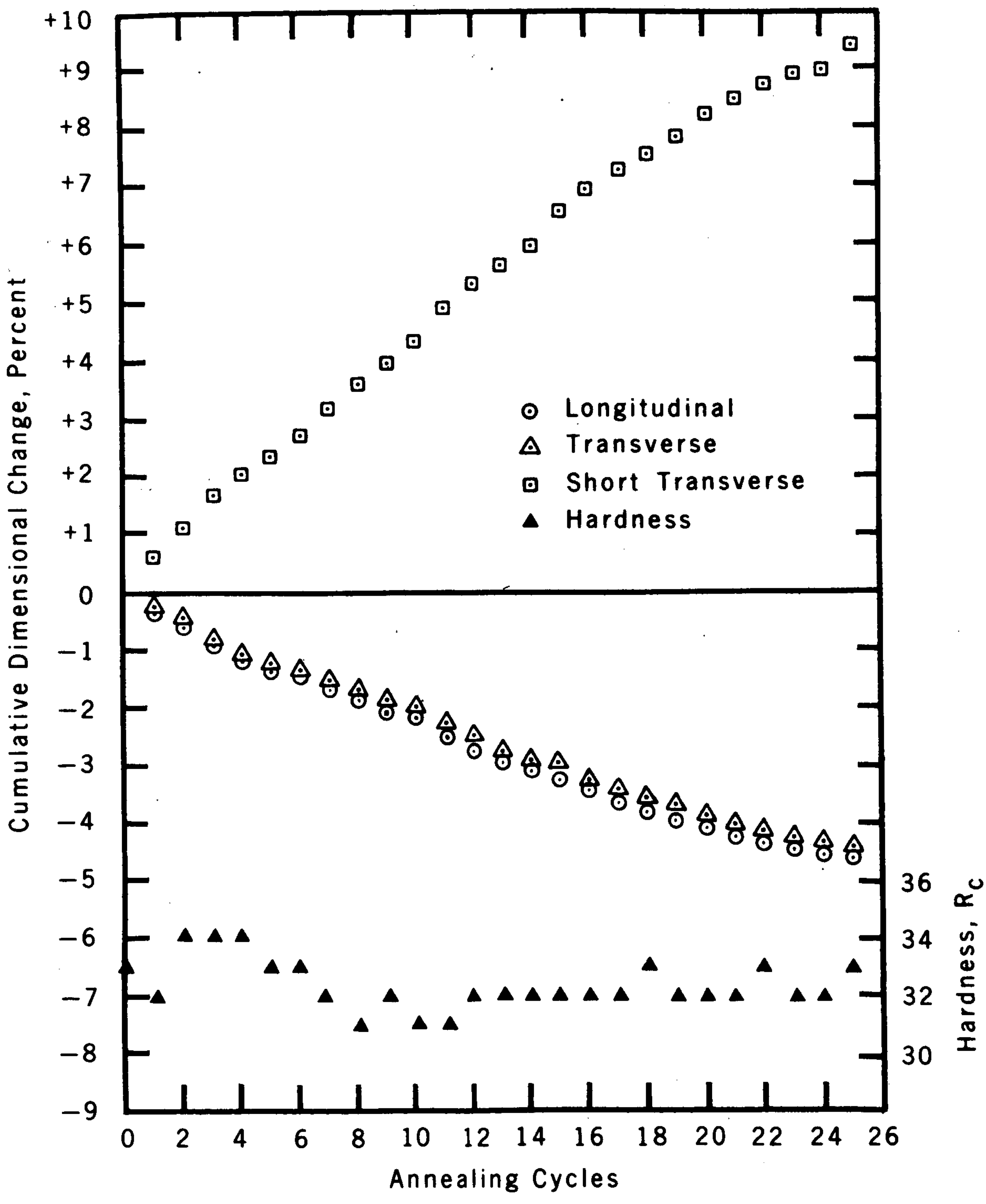


FIGURE 7 DIMENSIONAL AND HARDNESS CHANGES IN 18% NICKEL MARAGING STEEL SPECIMENS AFTER MULTIPLE ANNEALING CYCLES

(a) One Anneal

(b) Thirty-Five Anneals

FIGURE 8 MICROSTRUCTURE OF 18% NICKEL MARAGING STEEL

(500X)

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The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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