Experiments on Substructure in Iron

by

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ABSTRACT

The development of substructure by recovery annealing in several high purity irons and in an iron 3.5% chromium alloy was studied by optical metallography. The subgrain size was found to be essentially independent of tensile prestrain within the range of 5 to 30% strain, and no appreciable growth occurred until annealing temperatures causing partial recrystallization were reached. The subgrain size for the several irons was approximately 1 to 2 microns and that of the iron-chromium alloy slightly less. In partially recrystallized material, subgrain growth was observed in the unrecrystallized matrix.

Initial flow strength was evaluated in a zone-refined iron and in the same iron after treatment in pure wet hydrogen, as dependent on prestrain, and recovery annealing temperature. For the zone-refined iron, the yield strength after annealing at intermediate temperature was significantly higher than the prestrain stress, whereas the hydrogen-purified iron showed a lower yield stress for similar treatment. Hence, it is concluded that the strengthening of the zone-refined iron is to be attributed to interstitial-substructure interaction rather than to substructure as has been suggested by some earlier work reported in the literature.

The effect of grain size on flow strength was also studied, with results showing that interstitial-grain boundary interaction contributes importantly to the strength of iron.

Introduction

Considerable interest has developed in recent years in the possible influence of substructure on flow and fracture of metals. Parker and Washburn[1] found that by straining mild steel from 4 to 8% and annealing at temperatures of 427 to 690°C, the ultimate strength was increased to above that of the cold worked material. In some cases the recovered lower yield strength was greater than the prestrain stress, and in others it was less. The recovered yield strength was always greater than the initial yield point. These effects were attributed to a strengthening influence of substructure boundaries introduced during annealing and this conclusion has been widely accepted (see for example Ref. 2). However, when the evidence supporting this view is carefully examined, it is not clear that the data are free of question. Thus, it may be asked whether the results of Parker and Washburn have been influenced by quench-aging or strain-aging phenomena.

Since Parker and Washburn's early work, others have examined the possible role of substructures, with somewhat controversial conclusions. Washburn[3] introduced dislocation arrays into zinc crystals, and observed that the strengthening effect depended upon the temperature of annealing of the specimen containing such boundaries. A pair of 1° boundaries introduced by bending at the temperature of liquid nitrogen caused no strengthening if the crystal was heated only to room temperature, only a small strengthening on heating to 300°C, but considerable increase of yield stress (up to 50%) on heating to 400°C. Li[4] concluded from theoretical considerations that polygonized edge dislocation walls should not significantly increase flow strength. Meakin and Wilsdorf[5] experimentally observed that although subboundaries in single crystals of alpha brass occasionally offered definite impedance to the motion of gliding dislocations, this was not generally true. Michalak[6], in studies of hydrogen purified, zone-refined iron observed a continuous decrease in strength on annealing plastically strained samples in the recovery range. However, no substructure was detected.

In view of the confused evidence, further experimental study seemed desirable, and consequently the present study was made. In order to assess the results of past or current studies, it is desirable to attempt to establish what might properly be accepted as evidence of a strengthening role for substructure. Certainly, the introduction of additional dislocations and vacancies during deformation at temperatures below the recovery range leads to strengthening (strain-hardening), and until the density of these defects is lowered to the original level, by an annealing process designed to develop substructures, it is reasonable to expect residual strengthening, even when the defects are rearranged into small angle boundaries. Thus the exact role of substructure is difficult to assess. However, if the rearrangement into small angle boundaries leads to an increase of flow strength beyond that of the as-strained material, as some investigators have claimed (for example, Parker and Washburn[1]), then a strengthening effect of substructure cannot be questioned, unless an alternative source of strengthening can be suggested. In the case of iron, interstitial impurity atom-dislocation interaction[8] is an alternative strengthening mechanism.

EXPERIMENTAL PROCEDURE

A conventional method of introducing substructure into metals involves plastic straining followed by annealing below the recrystallization temperature range. The development of substructure in iron has recently been reviewed by Keh[9], who also reported additional experimental results using electron transmission microscopy. A cell-like structure, comprised of tangled dislocations, forms in iron plastically strained at room temperature. Annealing at temperatures above 400°C resulted in the formation of regular dislocation networks and a decrease in dislocation density. There was no significant subgrain growth during recovery at temperatures below that necessary to initiate recrystallization.

For most of the present investigation, the base material was a zone-refined iron which was obtained from Battelle Memorial Institute, where it had been prepared under the auspices of the American Iron and Steel Institute. This material received six passes in an alumina boat. The chemical analysis of this material is reported in Table I. However, preliminary experiments to characterize the dependence of substructure on prestrain, time and temperature were made with a vacuum-melted and cast electrolytic iron* and a similarly processed iron-3.6% chromium alloy. Since our early studies suggested important interstitial effects in the zone-refined iron[8], the zone-refined iron was purified by annealing in wet hydrogen to obtain a lower interstitial content. Pure hydrogen was obtained by diffusion through a palladium-silver coil and saturated with distilled water at room temperature. Treatment was prolonged to a time beyond which further lowering of the yield strength was not observed. Carbon was analyzed by the Fundamental Research Laboratory of United States Steel Corporation in the specimens after purification and reported as 13 ppm for the hydrogen-purified iron, as compared with 19 ppm for the non-purified material (Battelle analysis, 10 ppm, Table I).

Wire tensile specimens were prepared from the zone-refined iron bar by cold rolling and drawing. During preparation of the wire, great care was taken to avoid contamination; no lubricant was used and the wire was degreased with acetone between all stages of preparation and prior to annealing. After rolling to 100 mil. diameter rod (86% reduction of area), part of the zone-refined iron was wet-hydrogen-purified for 50 hours at 750°C to reduce the interstitial impurities, and part was annealed (for recrystallization) at 750°C in vacuum. The rods were cold drawn to 50 mil. diameter wire (75% reduction of area) and specimens 3 inches long were cut from the wire stock. To obtain uniform small grain size, the wire specimens were recrystallized at 625°C for 5 hours, in vacuum for as-received zone-refined iron and in wet hydrogen for hydrogen-purified iron. The resulting grain size of both materials was about 80-100 microns (ASTM 4-5). After prestraining, the heat treating of both materials was performed in thoroughly outgassed vycor capsules in a vacuum of about 10^{-5} mm Hg; the capsules were withdrawn from the furnace and cooled in still air.

Tensile tests were conducted at room temperature using an Instron testing machine. Specimens with a gage length of 1.60 inches between grips were loaded at a crosshead speed of 0.02 inches per minute, which gave an initial strain rate of 1.25×10^{-2} min.$^{-1}$. The yield strength was defined as the lower yield stress in zone-refined iron, for which a well-defined yield point was always observed. In some cases, small yield points were observed in the hydrogen-purified iron, but the maximum length of the yield was 0.1% strain in hydrogen-purified iron as compared to 2.7% strain for zone-refined iron. For hydrogen-purified iron, the yield strength was defined as the stress at 0.1% offset.

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*C, N and O contents of this iron were respectively 0.005, 0.001 and 0.031 per cent by weight.

EXPERIMENTAL RESULTS

Delineation of Substructure

A special metallographic technique was developed to reveal substructure in iron regardless of purity. Specimens were mechanically polished and then electropolished in a stirred solution containing 65 parts of orthophosphoric acid, 35 parts of ethyl alcohol and 35 parts water for 4 minutes at 1 volt D. C. It was discovered that an etchant developed by Gorsuch[10] for studying dislocations in iron whiskers revealed substructure by etching the subboundaries and decorating them by preferentially depositing a thin layer of copper at the dislocation wall. To avoid copper deposition over the entire specimen, the etching time was limited to one second, and the specimen was rinsed immediately under flowing water. The composition of the etchant was

2.5 gms picric acid
4.0 gms anh. cupric chloride
100 ml ethyl alcohol
10 ml hydrochloric acid
3 ml nitric acid

This etchant exhibits an orientation sensitivity, so that not all grains in a polycrystalline specimen can be etched to show substructure.

In vacuum-melted electrolytic iron with a carbon concentration of about 50 ppm, a picral etchant satisfactorily reveals substructure and is not orientation dependent, allowing the number of grains containing substructure to be determined.

Characterization of Substructure

To explore the effect of the strain and recovery temperature on the development of substructure in iron, a series of tensile specimens of vacuum-melted and cast electrolytic iron and an iron-3.6% chromium alloy (referred to earlier) were prepared and recrystallized at 840°C for two hours. For electrolytic iron the resulting grain size was about 500 microns, and for the iron-3.6% chromium alloy the grain size was about 250 microns. Specimens were strained in tension different amounts in the range 6 to 10%. Samples were cut from the gage section and annealed one hour at various temperatures in the range 600 to 835°C.

No significant variation in subgrain size in vacuum-melted electrolytic iron was observed with strains from 6 to 10% for constant annealing temperature. The subgrain size was approximately 1 to 2 microns for annealing at 600°C. Above 650°C partial recrystallization was observed and as the temperature was increased to 800°C subgrain growth occurred in the unrecrystallized grains. Figure 1 shows typical substructures observed in electrolytic iron.

For the iron-3.6% chromium alloy, the substructure developed after annealing at 600°C was slightly finer, 0.5 to 1.5 microns, than that observed in iron and not quite as well defined. Again, no dependency of subgrain size on strain was observed within the range of 6 to 10%. As the annealing temperature was increased from 700 to 835°C, partial recrystallization occurred and subgrain growth was observed in the unrecrystallized matrix. Typical substructures observed in the iron-3.6% chromium alloy are shown in Figure 2.

Since no significant difference in subgrain size was observed in the range of 6 to 10% strain in vacuum-melted electrolytic iron and iron-3.6% chromium, a wider range of 5 to 30% strain was studied in as-received and in hydrogen-purified, zone-refined iron wire. In initial experiments with zone-refined iron wire, the development of substructure was evaluated for strains of 10 and 30% and annealing temperatures of 350, 450 and 500°C for 16 hours. Figure 3 shows the progress of the development of substructure in the 30% specimen with increasing annealing temperature. Substructure appeared to form directly in the slip traces. During annealing the slip traces appeared to become more sharply defined and discrete cells became recognizable. In the 30% specimen, the cell size remained essentially uncoarsened even after annealing 16 hours at 500°C. Thus, the picture obtained of the nature of substructure in iron is a "perfecting" of the deformation structure (see also Ball[11] and Keh[8,9]). In general, the degree of definition of substructure was heterogeneous throughout the polycrystalline specimens; in the same specimen sharply defined cells were observed in some grains while other grains showed only slip traces.

In this fine-grained iron, recrystallization occurred simultaneously with the development of well-defined subgrains, and the specimens were largely recrystallized before significant subgrain growth occurred. The subgrain size was essentially independent of prestrain between 10% and 30% strain.

With 30% strain, recrystallization nucleation was observed after annealing 16 hours at 450°C. Substructure was best defined in the 30% specimen annealed at 500°C, where considerable recrystallization had already occurred elsewhere in the specimen. For the specimen strained 10%, higher annealing temperatures were required to obtain a degree of definition comparable to the 30% specimen, but the subgrain size was approximately the same for the same annealing temperature.

For subsequent study of the effect of substructure on flow strength of the zone-refined iron and of hydrogen-purified, zone-refined iron, prestrains of 5, 10, 15 and 25% were employed. To obtain maximum recovery and subgrain definition for this range of strain, without recrystallization, the specimens were annealed at 450°C for 4 hours. With this heat treatment, well-defined substructures were observed in the strained specimens 15 and 25%. However, in the specimens strained 5 and 10%, the substructure remained ill-defined after this annealing treatment. Well-defined substructure was observed in the 5% strain specimen only after annealing at 600°C, which brought about concurrent recrystallization nucleation.

The substructures observed in the hydrogen-treated and the nontreated zone-refined irons were essentially identical for the same conditions of strain and annealing below the recrystallization temperature.

Mechanical Properties

To evaluate the relative effects of interstitial impurities and dislocation substructures on flow strength, the tensile behavior of as-received and hydrogen-purified zone-refined iron was studied after a series of strain and recovery-anneal treatments. Annealing temperature was varied from 200 to 600°C, and for most of the study the holding time was four hours. The prestrain was varied from 5 to 25%. Results are tabulated in Table II for hydrogen-purified iron and in Table III for zone-refined iron.

Hydrogen-purification of zone-refined iron has a major effect on the stress-strain curve as shown in Figure 4, reducing the yield strength from 16000 to 6500 psi. As mentioned earlier, chemical analysis of these materials showed that treatment in hydrogen had reduced the carbon by 6 ppm. Over 50% of the yield strength of annealed zone-refined iron is therefore presumably attributable to the interaction of interstitials with dislocations. Hydrogen-purification also decreased the ultimate strength, from about 31,600 to 28,000 psi, increased the elongation, and increased the reduction of area at fracture (not reported in Tables II and III) from 53 to 69%. It is of interest that the zone-refined iron exhibited a sharp yield point, which extended to 2.7% strain whereas after hydrogen purification yield points less than 0.1% strain were observed.

Since yield strength data for single crystals of hydrogen-purified iron were available for the same test conditions from the work of H. H. Kranzlein[12], it is possible to differentiate the factors contributing to the measured strength of polycrystalline zone-refined iron. Figure 5 illustrates schematically the relative proportions of strengthening due to the intrinsic strength of zone-refined iron (yield stress of a single crystal), due to grain boundary strengthening, and due to interaction of interstitials with grain boundaries. In Figure 5, line A represents the yield strength of polycrystalline zone-refined iron, line B is the yield strength of polycrystalline, hydrogen-purified, zone-refined iron of the same grain size, and line C is the yield strength of a hydrogen-purified, zone-refined iron single crystal.* As can be noted from the figure, the effect of grain boundaries on strength at low interstitial concentrations is relatively small. In addition, no effect on strength was observed when unstrained specimens were annealed at temperatures up to 550°C as is indicated by the figure.

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*The yield strength of the single crystals did not vary appreciably with orientation within the range examined[12], owing presumably to the multiplicity of possible slip systems in iron.

After straining followed by annealing within the recovery range, hydrogen-purified iron exhibited a continuous decrease in yield strength with increasing temperature, as shown in Figure 6. Values for percentage recovery are given in Table IV. Even at 200°C where large increases in strength due to strain aging are observed for many irons and steels, more than 5% recovery was observed for the purified material that had been strained 5%. At 450°C, the maximum recovery temperature possible while avoiding recrystallization nucleation following 25% strain, the recovered yield strength was a function of prestrain, as shown in Figure 7, and was about 30% less than the prestrain stress. It is interesting that the ultimate tensile strength of the hydrogen-purified material remained essentially fixed throughout these annealing treatments (Table II and Fig. 12).

In contrast to the hydrogen-purified iron, a strong interstitial dislocation interaction (strain-aging) was observed in zone-refined iron after the strain-anneal treatments, as shown in Figure 8. After prestraining 5% and annealing at 200°C, the yield strength was 40% greater than the flow stress for 5% strain. For prestrains of 10, 15 and 25% the percentages of strain - aging (at 200°C) were 29, 24, and 18% respectively. The observed strain-aging was also reflected in an increase in ultimate tensile strength (Table II and Fig. 12).

If the data for hydrogen-purified iron are taken as a base, it is possible to separate, for a given prestrain and annealing temperature, the portion of strengthening due to interstitial-dislocation interaction from that intrinsic in iron. Table V presents values for the "inherent strength" and for the interaction strength obtained in this manner for zone-refined iron. Further lowering of carbon could be expected only to lower the inherent strength; hence the interaction strengths of Table V are minimal.

Figures 9 and 10 illustrate graphically the intrinsic strength, and dislocation-interstitial interaction strengthening of zone-refined iron after straining 5 and 10% and annealing. Although the contribution to the strength of zone-refined iron by interaction is reduced by annealing at the higher temperatures, this factor is still a major fraction of the strength observed at the highest temperature studied. As shown in Figure 11, the yield strengths of the 5 and 10% specimens after annealing at 450°C were still slightly greater than the prestrain stresses. For this same annealing temperature, the yield strengths of the 15 and 25% specimens were slightly less than their prestrain stresses. The levelling may be explained in terms of the saturation of interaction strengthening due to insufficient interstitial impurities, the relatively small effect of unpinned dislocations on strength, and to faster recovery at higher strains.

The variation with prestrain of ultimate tensile strength of the zone-refined and hydrogen-purified irons is shown in Fig. 12 for several annealing temperatures. Unfortunately data are not available for large prestrains and annealing temperatures of 200 and 550°C for the hydrogen-purified iron. Also the datum for this iron for the prestrain of 10% and annealing at 200°C represents 1 hour, rather than 4 hours, as do the other data. However, it is reasonable to assume no difference in yield strength for 1 to 4 hours.

(See e.g. data in Table III for 1 and 4 hours at 200°C for zone-refined iron)

Discussion of Results

Investigators have generally tended to overlook the possibility of "strain aging" in iron after higher temperature anneals (above perhaps 300°C). In some cases, the high yield strength observed after annealing just below the recrystallization temperature in iron has been attributed to the formation of substructure[1]. However, the present results suggest that important interstitial-dislocation interaction strengthening may persist in iron to relatively high temperatures (see e.g. Fig. 9) Moreover, the present results as well as other results reported in the literature have shown that the sharpening of substructure in iron by recovery annealing results in a decrease in strength. This has been shown in the present investigation by two irons which with similar substructures but different interstitial impurity contents show completely opposite behaviors. For hydrogen-purified iron, recovery of yield strength was observed after prestrain anneal treatments; moreover, no increase in the ultimate strength was observed. However, for as-received zone-refined iron, identical pre-treatments resulted in substantial increases in both the yield strength and the ultimate strength. Thus, the ultimate tensile strength of the zone-refined iron was increased from 31,600 to 38,200 psi by prestraining, 10-25%, followed by heating at 200°C.

The mechanical behavior of iron containing interstitial impurities after prestrain and annealing treatments must be considered as the net result of two processes. When a strained specimen is annealed below the recrystallization temperature, recovery occurs by the straightening, annihilation and rearrangement of dislocations and vacancies. With increasing prestrain, the cells became more sharply defined even for room temperature deformation.[13] Increasing the annealing temperature causes sharpening and perfecting of the cell structure developed during deformation. However, in polycrystalline iron, recrystallization begins before significant subgrain growth can be obtained throughout the entire specimen. The formation of well-defined substructures by annealing is associated with a decrease in strength. Concurrent with recovery, interstitial impurity atoms associate with the new dislocations introduced by the straining, with a resultant strengthening*.

The net effect of the softening and strengthening processes may be expected to depend upon the dislocation density, the concentration of interstitial impurities, and the annealing temperature. The present results show that interaction strengthening is greatest at about 200°C, but that interaction makes an important contribution to flow strength, even for fairly high annealing temperatures. Furthermore, it is unjustified to look upon the formation of substructure, in the absence of impurity interactions, as causing strengthening. To the contrary, as the substructure becomes "perfected", flow strength progressively decreases.

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*The nature of the association is presumably that of the Cottrell "cloud" at low annealing temperatures[14], but might be one of precipitate particles at high annealing temperatures.

It is of interest that the analysis of the zone-refined iron before and after treatment in pure wet hydrogen showed that the carbon content had been reduced from 19 to 13 ppm. It is difficult to understand how such a reduction could cause such large change in the flow curve of the annealed iron, as well as change in the response to recovery annealing

That polygonization boundaries do not provide an important strengthening influence in the absence of interstitial impurities is also suggested by the results of a related research program concerned with the influence of grain size on the flow strength of the same materials, some results of which are summarized in the Petch type of plot, Figure 13. The slope of the plot of yield strength at room temperature versus the reciprocal of the square root of the grain size for the hydrogen-purified iron of the present study (0.381 Kg $mm^{-3/2}$) is the lowest of which we are aware in published literature, whereas, complex interaction is indicated in the non-hydrogen-treated iron. If large angle boundaries have so little effect on strength of iron (see also Fig. 5) it seems unreasonable to expect a small angle polygonization boundary to be an important barrier.

It also seems noteworthy that the yield strength of single crystal iron does not appear to be substantially different for the hydrogen-purified material or the unpurified material, suggesting that the interaction of interstitials with randomly dispersed dislocations at least in the densities prevalent in the materials of this study is not an important strengthening influence, in contrast to the situation as regards either small or large angle dislocation boundaries.

SUMMARY

1. Subgrain size produced in high-purity iron by recovery annealing is essentially independent of tensile prestrain within the range 5 to 30 per cent strain.

2. Substructure appeared to develop along markings that formed during deformation, and sharpened with increase of annealing temperature.

3. The substructures observed in the hydrogen treated and the non-hydrogen treated irons were essentially identical for the same strain and annealing conditions.

4. No appreciable growth of subgrains was observed until annealing temperatures causing partial recrystallization were reached, whereupon coarsening occurred.

5. The subgrain size for annealing temperatures not causing coarsening was about 1 to 2 microns.

6. A large fraction of the flow strength of the recrystallized, zone-refined iron of this investigation is attributable to interaction between interstitial impurity atoms and grain boundaries.

7. Considerable strengthening attributable to interstitial substructure interaction was observed in zone-refined iron containing 19 ppm of carbon, by analysis, when plastically strained and annealed in the recovery range.

8. Zone-refined iron in which carbon had been reduced from 19 to 13 ppm by treatment in wet hydrogen, showed progressive softening on annealing after plastic straining, even though the substructure appeared to be identical with that of iron not treated in hydrogen.

9. The rearrangement of dislocations into increasingly perfect subgrains by annealing in the recovery range results in a progressive decrease of yield strength from the level reached during prestraining, when the interstitial content is reduced to low level, leading to the conclusion that substructure does not make an important contribution to the strength of iron in the absence of interstitial-dislocation interactions. This conclusion is supported by the observation that the yield strength of the hydrogen treated iron is not greatly different in the mono- and polycrystalline conditions, indicating that large angle boundaries are not important barriers.

10. The similarity in strength of single crystals of zone-refined iron, whether hydrogen-purified or not, suggests that interaction of interstitial impurity atoms with dispersed dislocations is not an important strengthening influence for the dislocation densities and interstitial contents here studied.

11. The present results suggest that studies of recovery of iron, after plastic deformation are likely to be confused by interstitial-dislocation interactions.

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TABLE I

ANALYSIS OF ZONE-REFINED IRON

(As reported by Battelle Memorial Institute)
Amounts are given as parts per million
(1 ppm equals 0.0001 per cent)

Aluminum	15	Magnesium	5
Antimony	5 ND (a)	Manganese	0.5
Arsenic	10 ND	Molybdenum	5 ND
Beryllium	0.2	Nickel	12
Boron	5	Nitrogen	2
Calcium	10 ND	Oxygen	17
Cadmium	5 ND	Phosphorus	5
Carbon	10	Silicon	10
Chromium	5	Sulfur	9
Cobalt	1	Tin	5 ND
Copper	2	Titanium	0.5 ND
Hydrogen	2	Tungsten	10 ND
Lead	1 ND	Vanadium	1 ND
		Zinc	10 ND
		Zirconium	0.5 ND

(a) ND means not detected. Detection limits are given.

TABLE II

EFFECT OF PRESTRAIN-ANNEAL TREATMENTS ON THE MECHANICAL PROPERTIES OF HYDROGEN-PURIFIED IRON

Prestrain %	Initial Yield Strength psi	Prestrain Stress psi	Annealing Temp. °C	Annealing Time hr	Recovered Yield Strength psi	Ultimate Strength psi	Total Elongation %
0	6400					28,300	51.6
0	6500					27,600	48.0
4.9	7500	18,900	200	4	17,900	25,500	19.5
5.0	7000	18,700	200	4	18,100	26,400	23.4
5.0	6700	18,600	450	4	14,800	27,800	55.7
5.0	6800	19,100	450	4	15,600	28,100	53.6
4.9	7500	19,400	550	4	14,700	27,600	38.0
5.1	7000	18,900	550	4	14,000	27,500	36.4
10.0	6500	22,900	200	1	22,100	27,500	47.2
9.8	7100	22,700	450	4	17,600	26,500	47.1
9.9	6600	23,200	450	4	17,700	27,500	53.9
14.9	6700	25,300	450	4	19,200	26,700	49.6
15.0	6600	25,200	450	4	19,100	27,000	51.9
24.8	6400	27,000	450	4	19,900	26,400	33.4
23.0	6600	26,800	450	4	20,500	27,900	57.1

TABLE III

EFFECT OF PRESTRAIN-ANNEAL TREATMENTS
ON THE MECHANICAL PROPERTIES OF
ZONE-REFINED IRON

Prestrain %	Initial Yield Strength psi	Prestrain Stress psi	Annealing Temp. °C	Annealing Time hr.	Recovered Yield Stress psi	Ultimate Strength psi	Total Elongation %
0	13,700					31,300	40.5
0	15,900					31,900	35.8
4.8	14,000	20,600	200	4	28,900	33,800	36.3
4.9	13,700	20,300	200	4	28,000	31,900	35.9
5.0	14,000	20,300	450	4	23,300	32,300	36.4
5.0	14,000	19,800	450	4	22,500	31,700	37.7
4.9	14,000	20,100	550	4	21,100	30,900	29.7
5.0	14,000	20,000	550	4	20,600	30,700	37.5
4.9	14,400	20,100	600	4	19,600	*	*
5.0	13,700	20,300	600	4	19,100	*	*
9.6	15,300	27,200	200	1	34,100	38,100	28.5
9.7	17,300	27,100	200	4	34,700	38,400	28.8
9.9	13,800	25,200	450	4	26,500	33,300	31.6
9.7	13,800	26,000	450	4	27,300	33,500	36.2
14.8	15,000	29,200	200	4	36,200	38,800	29.4
14.8	14,000	28,100	450	4	27,000	32,900	34.9
14.7	14,500	28,100	450	4	28,600	33,100	28.8
24.6	16,100	32,000	200	4	37,800	38,300	32.9
24.6	14,000	29,100	450	4	27,800	31,600	40.4
24.8	13,800	29,200	450	4	28,300	31,600	35.1

*Failed at the grip.

TABLE IV

FLOW-STRESS RECOVERY OF HYDROGEN-PURIFIED IRON

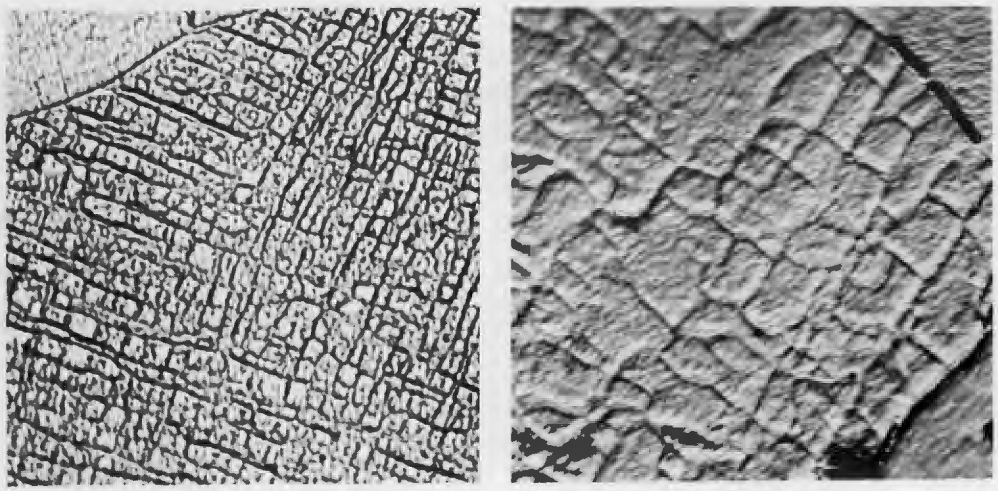
Prestrain	Annealing Temp. °C	Annealing Time hr.	Recovery of Flow Stress %
4.9	200	4	8.8
5.0	200	4	5.3
5.0	450	4	31.9
5.0	450	4	28.4
4.9	550	4	39.5
5.1	550	4	41.2
10.0	200	1	4.9
9.8	450	4	32.7
9.9	450	4	33.1
14.9	450	4	32.8
15.0	450	4	32.8
24.8	450	4	34.7
23.0	450	4	31.2

TABLE V

Analysis of Strength of Zone-Refined Iron

Prestrain	200°C Anneal		450°C Anneal	
%	Inherent Strength* psi	Interaction Strength psi	Inherent Strength* psi	Interaction Strength psi
5	18,000	10,400	15,200	7,700
10	22,100	12,600	17,600	9,300
15			19,200	8,500
25			20,200	7,800

*The "inherent" strength is here taken as the sum of the single crystal strength, and the grain boundary and residual strain hardening contributions, all measured for the hydrogen-purified material.

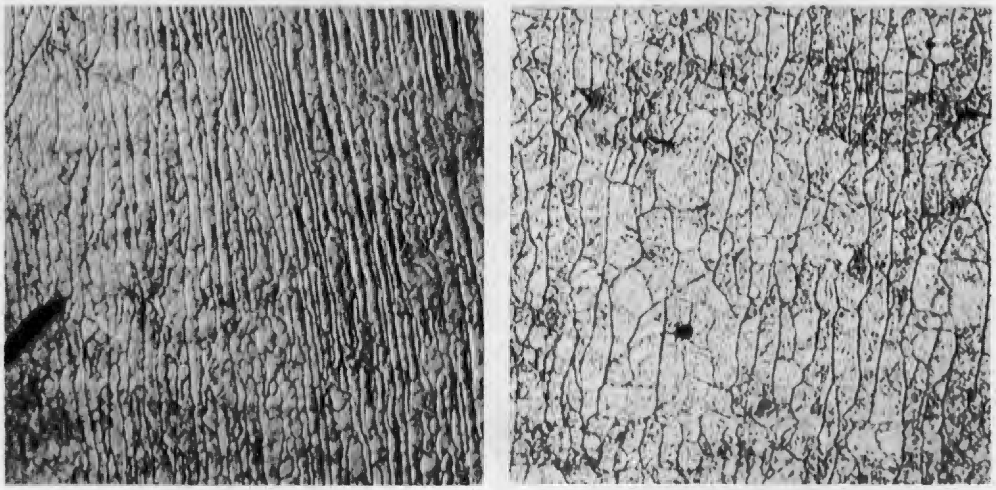


6% strain
600°C - I hr.

6% strain
800°C - I hr.

Substructure in vacuum-melted electrolytic iron; Gorsuch etchant; x 1000

Fig. I

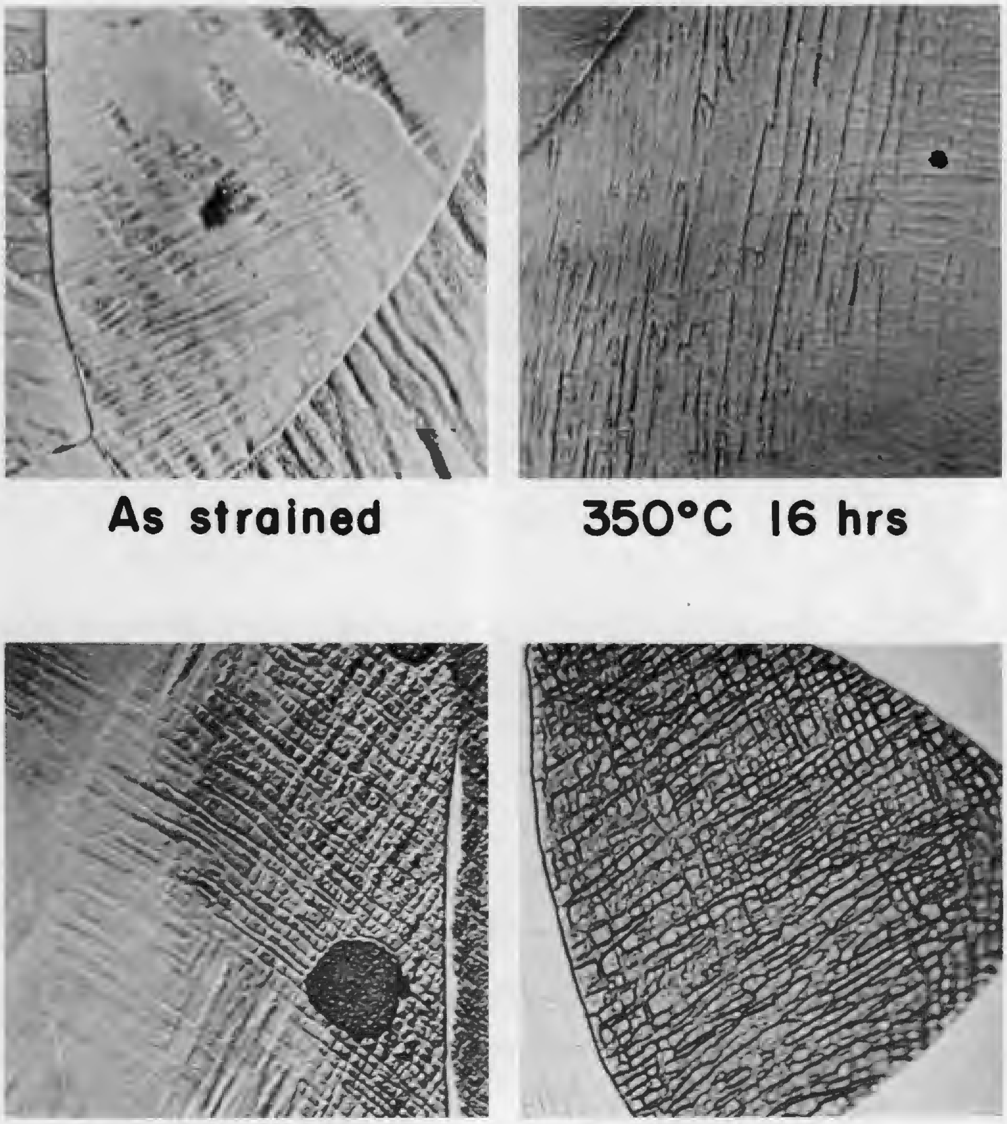


6% strain
600°C - 1 hr

8% strain
775°C - 1 hr

Substructure in vacuum-melted iron-3.6 % chromium alloy; Gorsuch etchant ; x 1000

Fig. 2



As strained — 350°C 16 hrs

450°C 16 hrs — 500°C 16 hrs

Substructure development in zone refined iron; 30 % strain; Gorsuch etchant; x 1000

Fig. 3

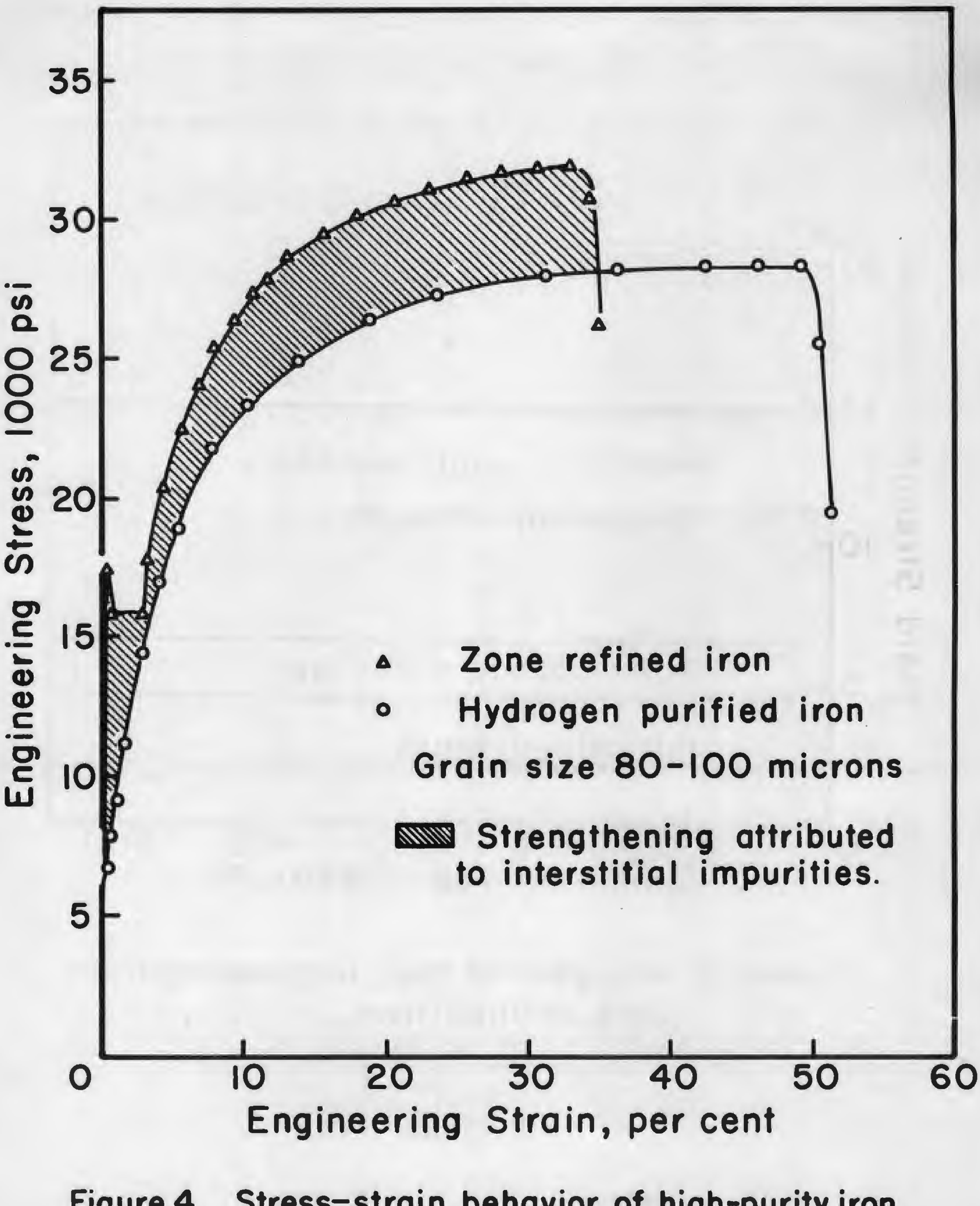


Figure 4. Stress–strain behavior of high-purity iron.

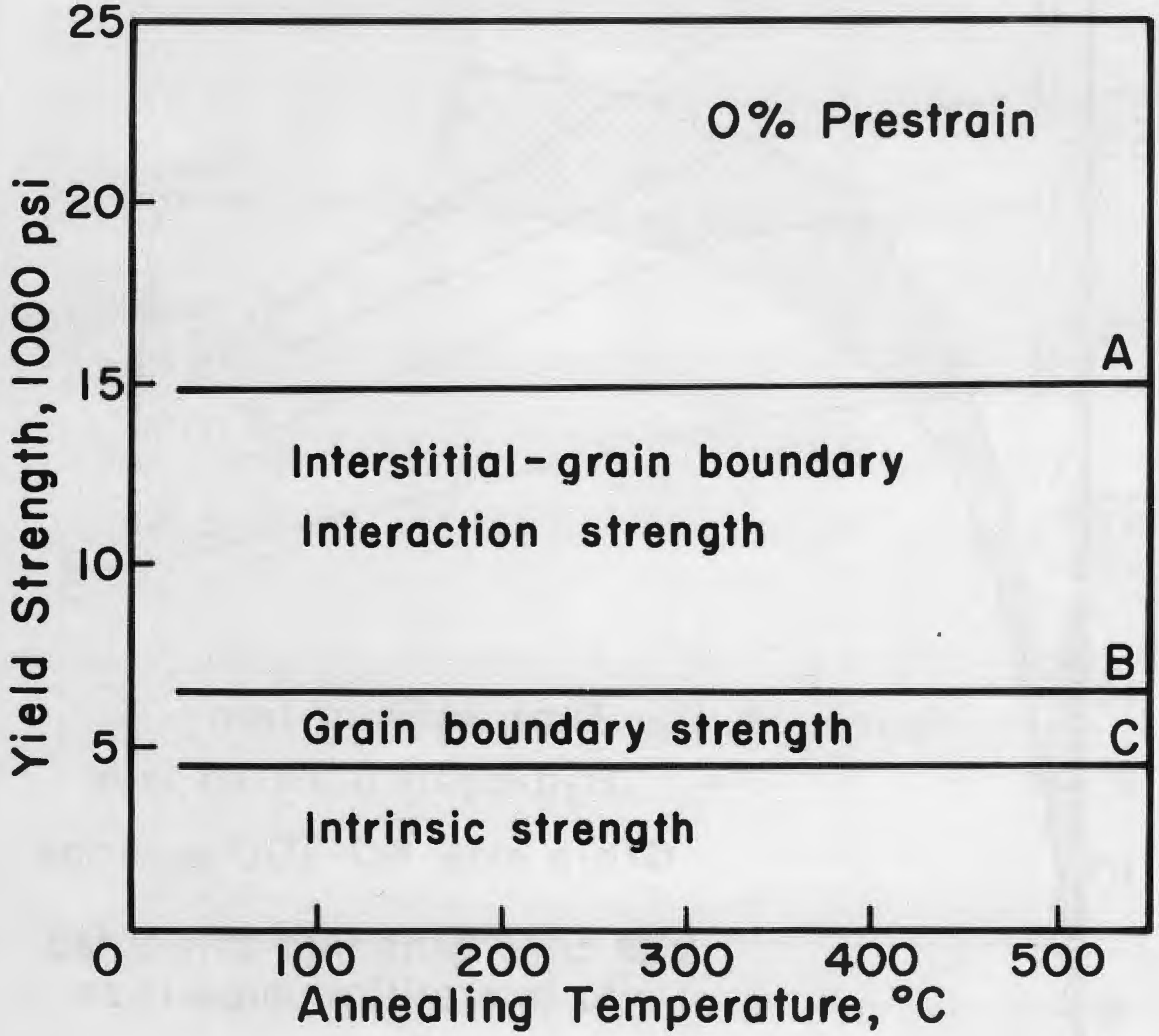


Figure 5 Analysis of the yield strength of zone refined iron.

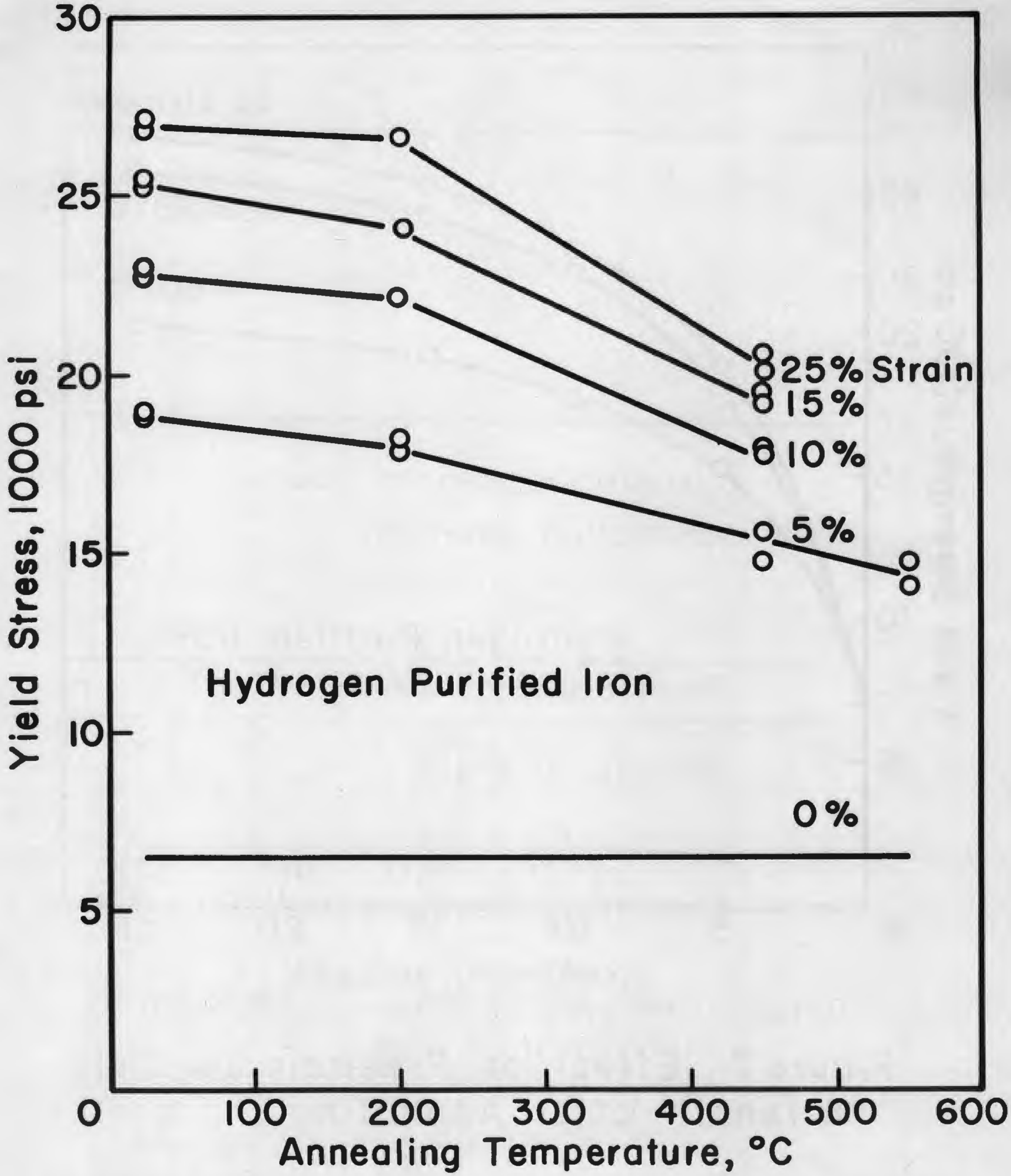


Figure 6 Effect of straining and annealing on yield strength.

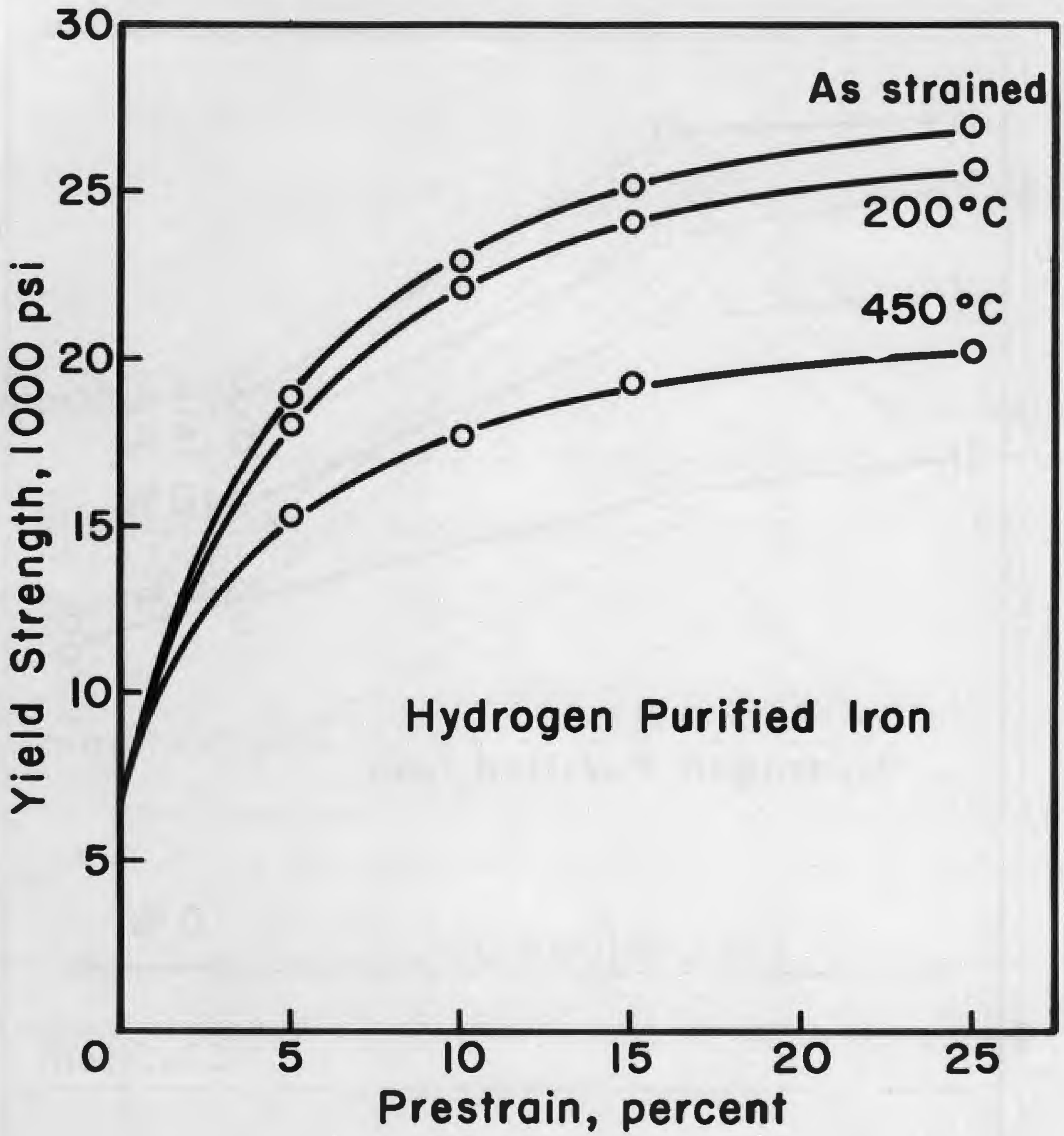


Figure 7 Effect of Prestrain on Yield Strength after Annealing

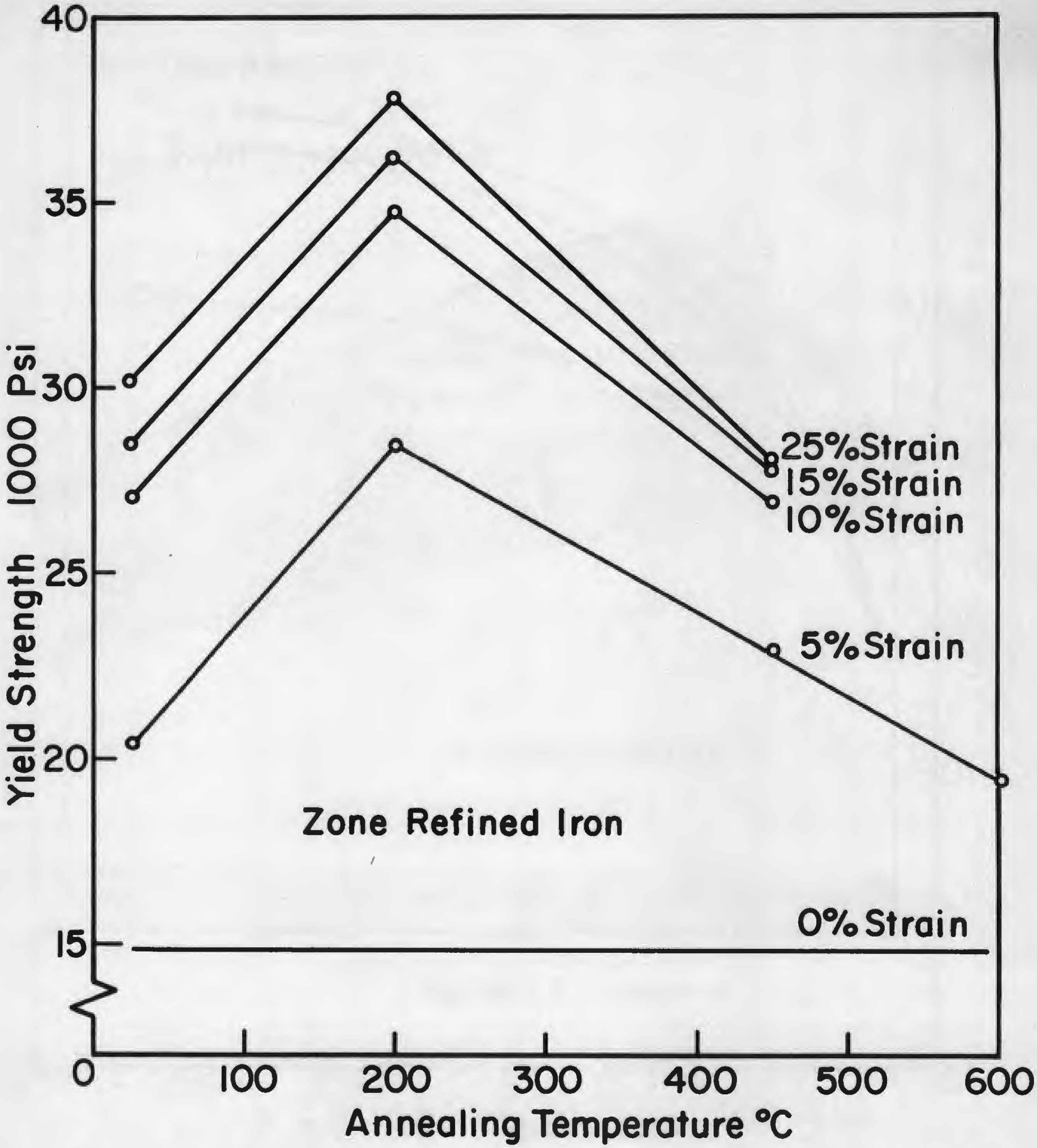


Figure 8. Effect of straining and annealing on yield strength.

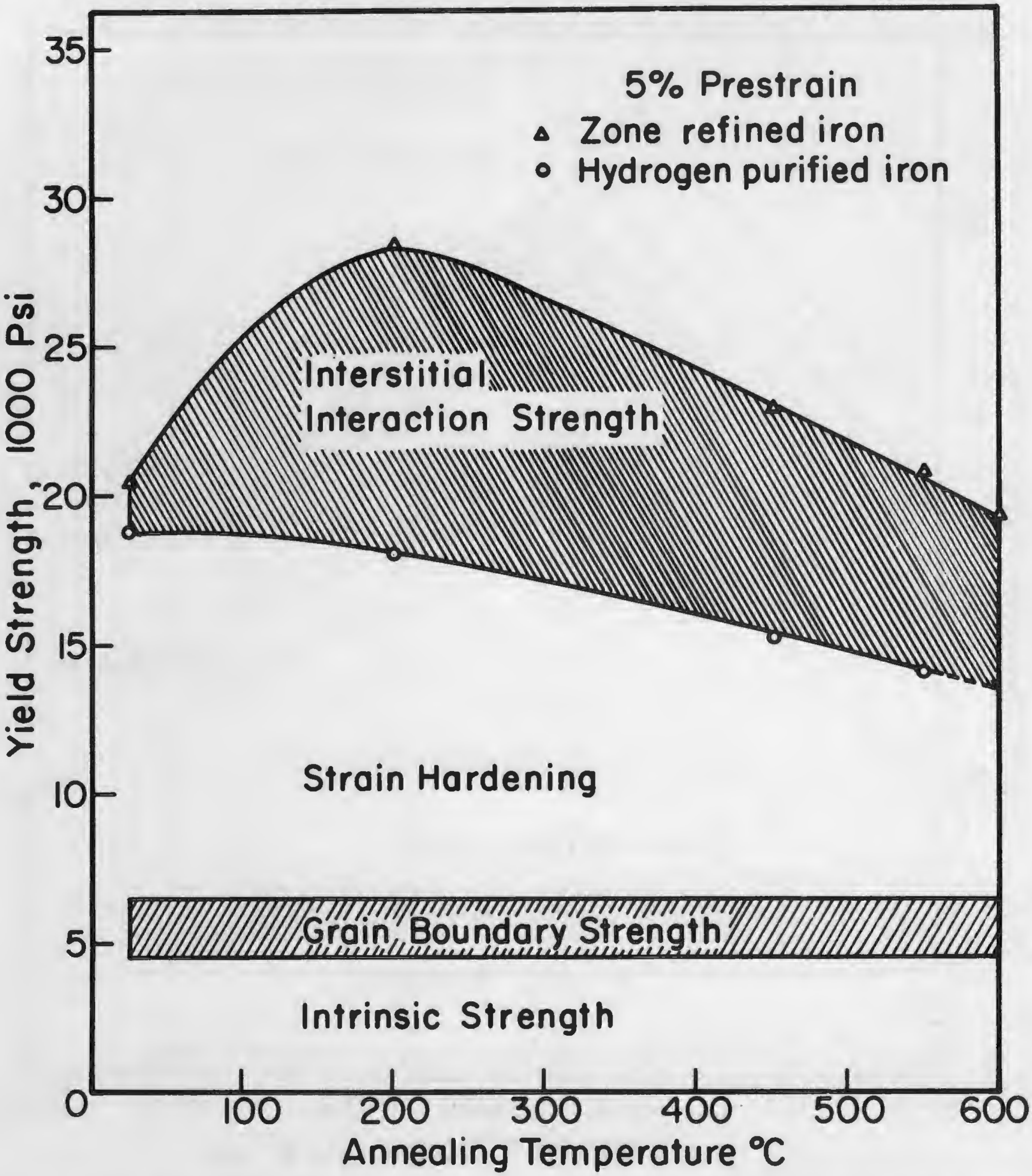


Figure 9. Analysis of yield strength of zone refined iron after strain-anneal treatments

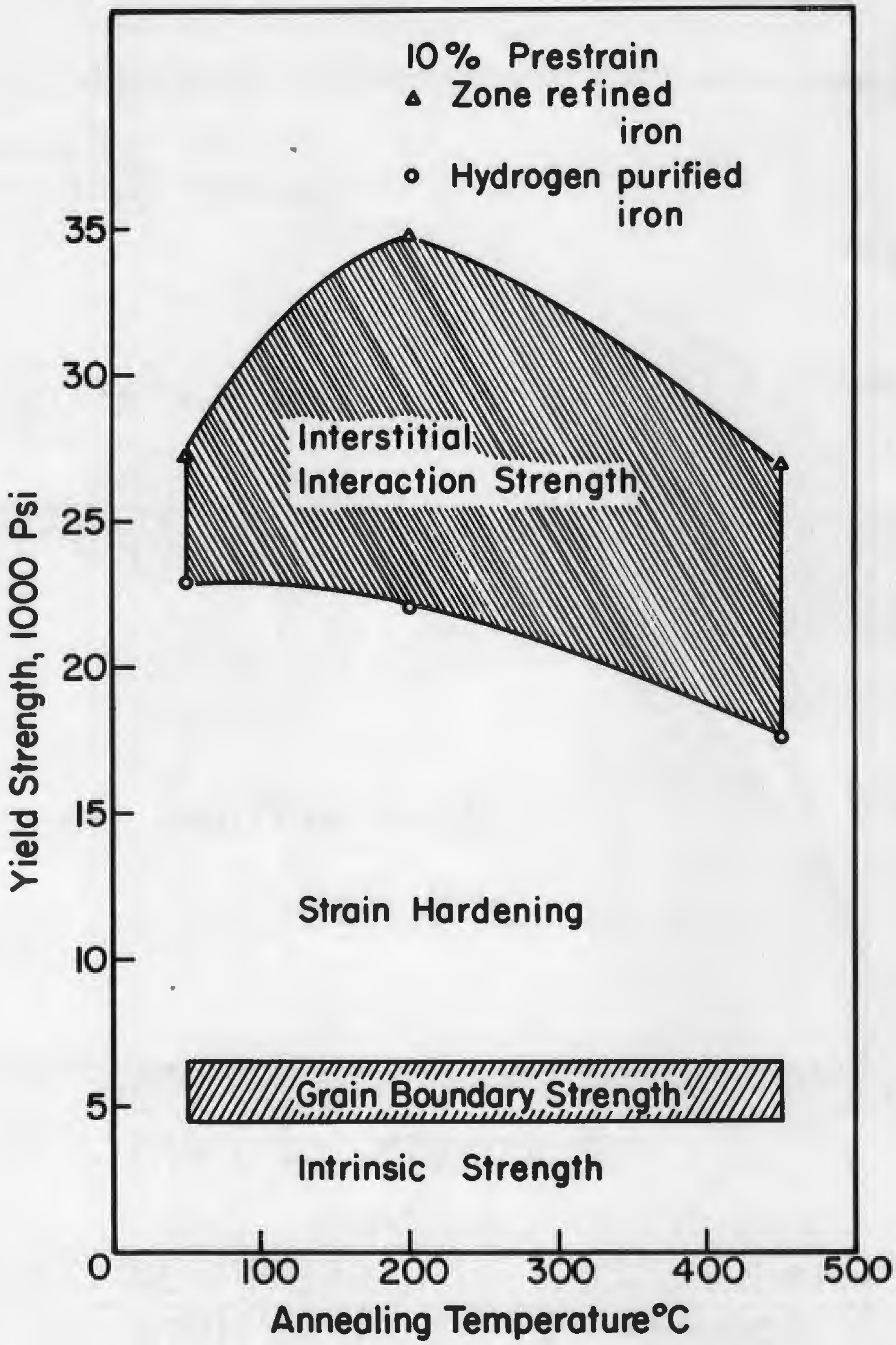


Figure 10. Analysis of yield strength of zone refined iron after strain-anneal treatments.

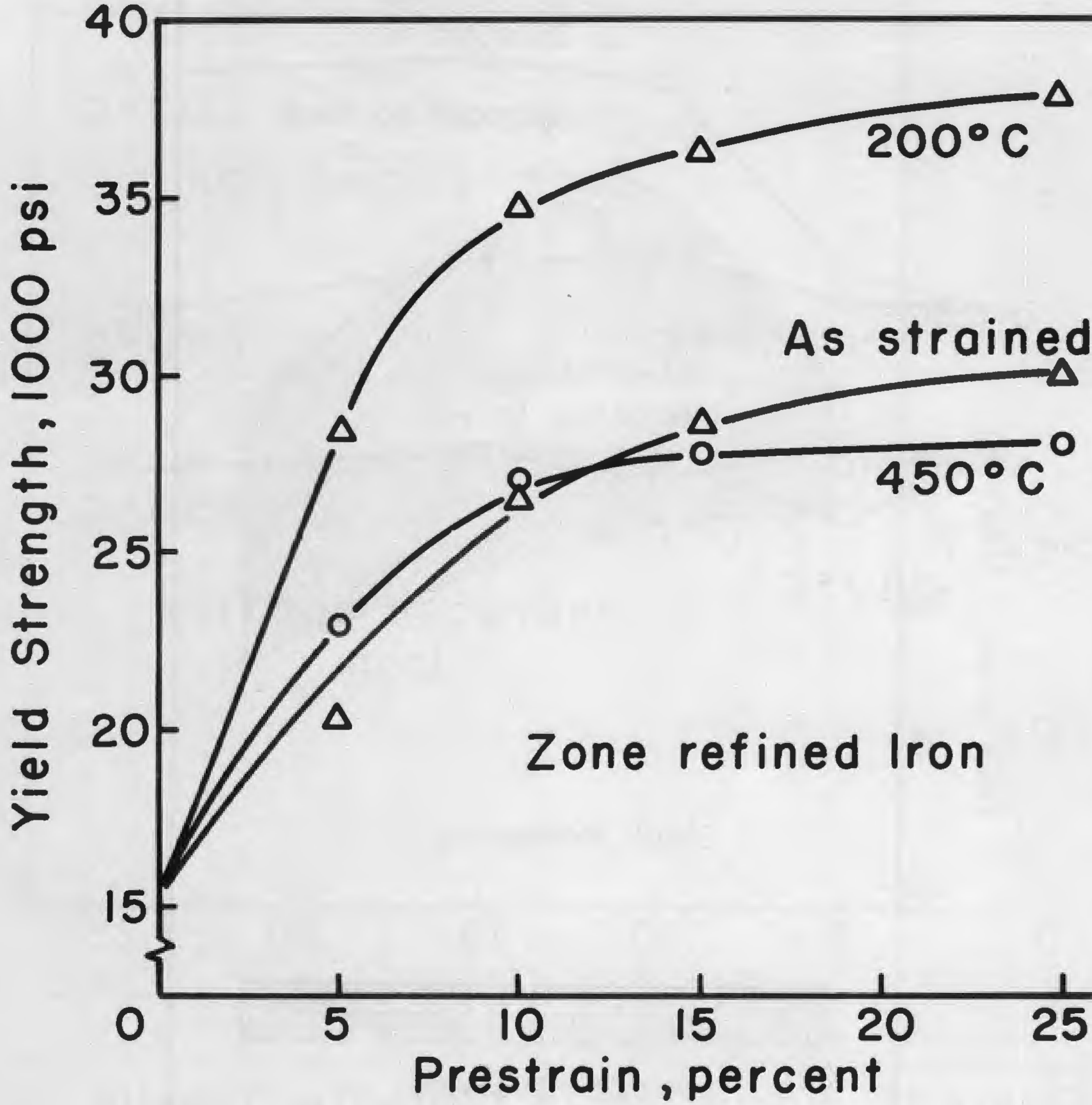


Figure 11 Effect of Prestrain on Yield Strength after Annealing

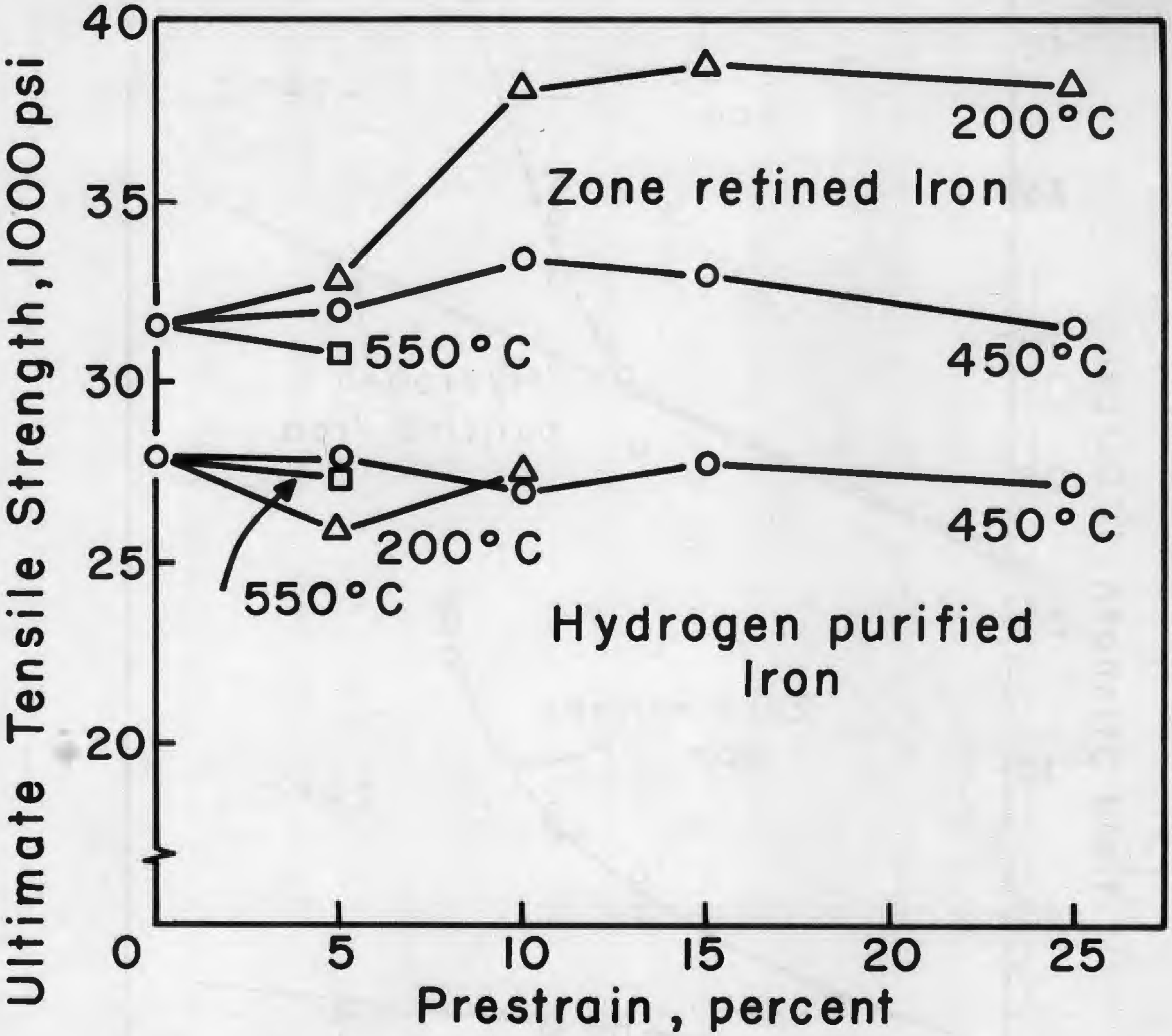


Figure 12 Variation of Ultimate Tensile Strength with Prestrain and Annealing Temperature

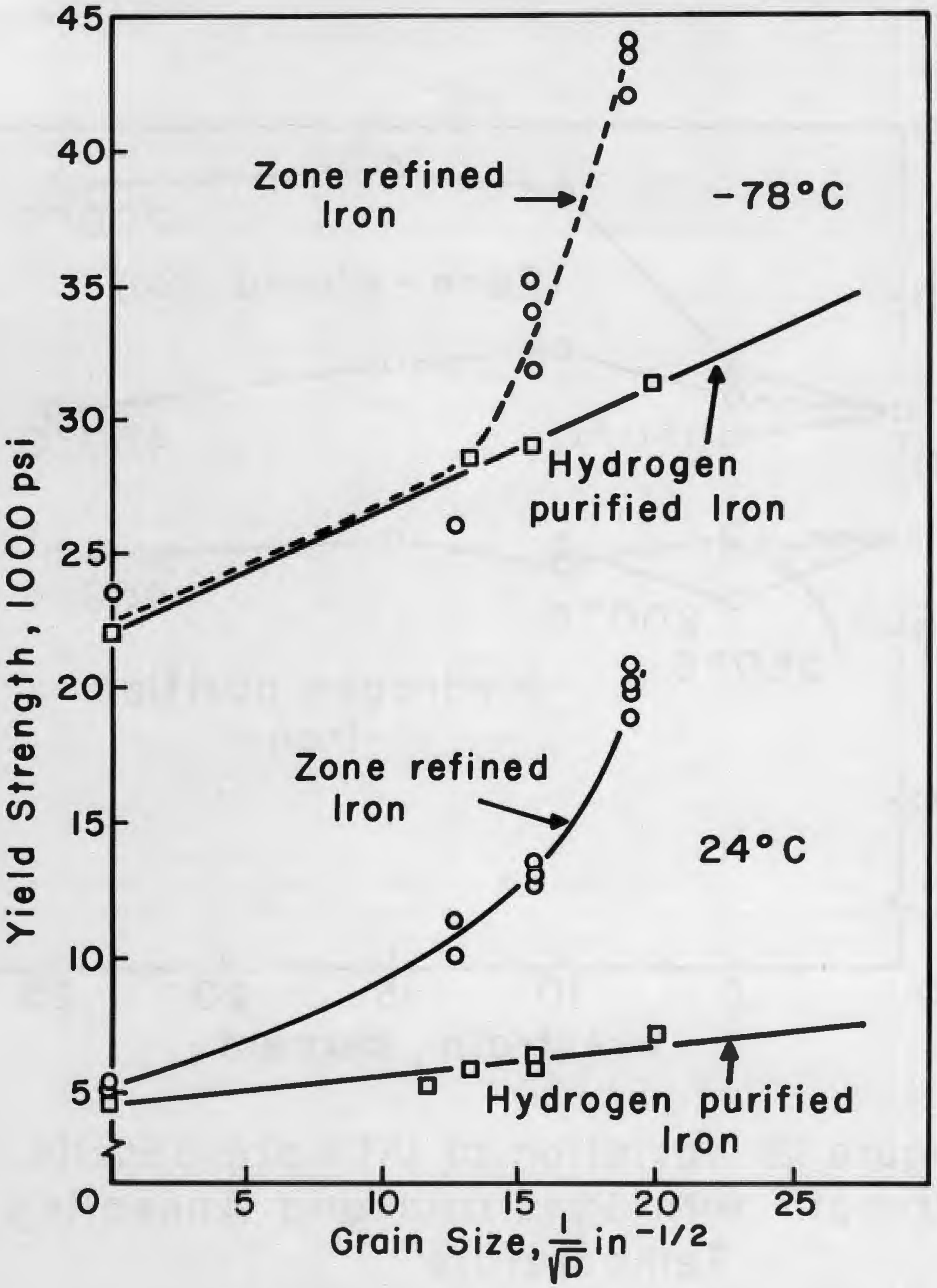


Figure 13 Variation of Yield Strength with Grain Size