EFFECT OF COLD-WORK ON AGING CHARACTERISTICS

OF BETA C Ti ALLOY

BY

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ABSTRACT

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OF BETA C Ti ALLOY

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The Beta-C (Ti-3Al-8V-6Cr-4Zr-4Mo) titanium alloy was solution treated and aged at two temperatures for several length of times to determine its hardening potential. Then the cold work alloy was also aged at the same temperatures and times, and the hardness profiles were studied for both cases. The cold worked alloy aged faster and to a higher hardness than the one which was only solution heat-treated and aged.

ACKNOWLEDGEMENT

I would like to thank my Heavenly God made this project completed successfully.

I would also like to thank Dr. Richard W. Jones, Director, Department of Materials Engineering, for his technical guidance and help throughout the studies.

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CHAPTER 1

INTRODUCTION

Several previous research studies indicate Beta-C titanium alloy is a good candidate to substitute for some of the other beta alloys due to its fewer fabrication problems, equivalent or superior mechanical properties and higher hardening potential [1]. The purpose of this study is to investigate the effect of cold-work on the hardness of aged Beta-C titanium alloy.

1.1 Age Hardening:

The principal methods for increasing the strength and hardness of alloy are as follows [2]:

1. Solid solution strengthening
2. Cold working
3. Heat treatment

The most important heat-treating process for nonferrous alloys is age hardening, or precipitation hardening [3]. In order to apply this heat treatment, the equilibrium diagram must show partial solid solubility, and the slope of the solvus line must be such that there is greater solubility at a higher temperature than at a lower temperature. These conditions are shown in Fig. 1. Consider the left side of the phase diagram involving the α solid solution. Alloy compositions that can be age hardened are usually chosen between point F containing 20% B and point H containing 10% B. Most age-hardenable alloys are chosen of compositions slightly to the left of point F, although the maximum hardening effect would be obtained by an alloy containing 20% B.

There are four steps in age-hardening heat treatment [4].

1. Solution Treatment: If the alloy 85A-15B (Fig. 1, and sketch of microstructure shown in Fig. 2) is heated to a temperature above the solvus temperature to point M and held long enough, a homogenous solid solution α is produced. This step dissolves the θ phase and reduces any segregation present in the original alloy.

2. Quench: After solution treatment, the alloy, which is single phase alpha, is rapidly cooled, or quenched. The atoms do not have time to diffuse to potential nucleation sites and permit the θ phase to form. After the quench, the structure remains single phase. The single phase is a supersaturated solid solution, containing excess B, and is not an equilibrium structure.

3. Aging: The supersaturated alpha is heated to a

temperature below the solvus temperature. At this aging temperature, B atoms are able to diffuse short distances. Because the supersaturated α is not stable, the extra B atoms diffuse to numerous nucleation sites and a precipitate forms. Eventually, if the alloy is held for a sufficient time at the aging temperature, the equilibrium $\alpha + \theta$ structure is produced.

4. Finally, the aged alloy (now hardened) is cooled to room temperature.

The process of precipitation is shown schematically in Fig. 3 [5]. Initially, B atoms segregate to form thin plates, referred to as Guinier-Preston (GP) zones. These thicken into plates in which B and A layers alternate, θ'' structure. As the θ'' precipitates increase in size, their crystal structures change to give θ' precipitates (shown by the square array in Fig. 3d). This stage usually corresponds to maximum hardness, and the precipitate is not resolvable through optical microscope. The θ' precipitate still has an interface on which atom positions match those in the matrix, giving a coherent interface. The match is not perfect, however, and as the θ' increases in size, the elastic strains at the interface increase, eventually attaining a magnitude to generate a complex dislocation network. The strain is relieved and the interface becomes incoherent. As θ' continues to grow its crystal structure and chemical composition changes to that of θ. Larger θ particles grow in size and continue to grow as smaller θ particles dissolve and the structure coarsens. This causes the strength to decrease.

The effect of aging temperature and time on the yield strength is shown schematically in Fig. 4. At the low temperature (T_1) the diffusion rate is so slow that no appreciable precipitation occurs. At T_3, hardening occurs quickly, due to rapid diffusion, but softening effects are also accelerated, resulting in a lower maximum hardness. The optimum temperature seems to be T_2, at which the maximum hardening occurs within a reasonable length of time.

1.2 Alloy Concepts:

Titanium has a hexagonal close packed (H.C.P) crystal structure at room temperature. This structure is called the alpha phase, and it is stable up to about 882°C (1620°F), where the structure changes to body-centered cubic. The minimum temperature above which the alloy exists entirely as the beta phase (body-centered cubic) is the beta transus temperature. Alloying elements provide solid solution strengthening and change the allotropic transformation temperature. Alpha grades of titanium are pure titanium that is solid solution strengthened by small additions of elements such as Al, Sn, O and N. Alpha stabilizing elements increase the temperature at which alpha transforms to beta. Beta alloys are produced by adding large amounts of beta stabilizing elements such as Mo, V, Ta and Nb to make beta the stable phase at room temperature [6]. At high levels of beta stabilizers, metastable beta can even be achieved by air cooling [7].

There are two types of beta stabilizers: beta isomorphous and beta eutectoid. Examples of alloying elements in the first class are Mo, V, Ta and Nb and in the second class Cr, Mn, Fe, Co, Ni and Cu. Schematic phase diagrams for the two classes of beta stabilizers are shown in Fig. 5. The terminal solid solution phase, alpha, and an enriched beta phase result from the decomposition of the metastable beta in the first case. In the second case beta decomposes into an eutectoid mixture of alpha and a compound. Active eutectoid formers (e.g.,Ni,Cu) promote rapid decomposition of beta phase to produce a compound and the α phase. Slow eutectoid formers (e.g., Fe, Mn) induce a slower decomposition of beta [6].

The effect of the age hardening heat treatment depends on such factors as chemical composition, heat treatment time and temperature, and cold work. Beta alloys have good ductility and formability when they are not age hardened. After heat treatment, the alloy has high strength but ductility and toughness are reduced. A brief description of the types of phases which form in meta-stable Beta-C titanium alloy is given below [8].

Beta, ß : High temperature allotropic phase of titanium and its crystal structure is BCC.

Beta Prime, ß′ : A metastable phase which forms in solute rich metastable Beta-C titanium alloy [9] where the omega phase formation is suppressed. The formation of ß′ reaction is known as phase separation (ß ⟶ ß′ + ß) and this coherent with beta.

Omega, ω : A metastable phase which can form when titanium is alloyed with elements which stabilize the high temperature b.c.c. structure (ß-phase). It is always metastable with respect to the equilibrium (ß + α) structures.

The various types of phase transformation and relative stability of the phases are shown in Fig. 6.

1.3 Heat Treatments:

The solution treatment consists of heating the alloy above beta transus to about 816-818°C and keeping at temperature for about 1 to 2 hours, followed by air cooling. This results in beta phase and unidentified second phase particles which have low strength and very high ductility. The strength of the alloy can be increased by aging treatments. There are two types of aging treatments and these are: high temperature aging and low temperature aging.

The high temperature aging consists of heating the alloy to about 85-195°C (150-350°F) below the beta transus for a short time (normally less than 24 hours), followed by air cooling. The higher the temperature, the coarser the alpha particles.

Low temperature aging is normally done in the temperature range of 200-450°C. The following phase transformation can occur, depending on the aging time and temperature:

$$\beta \longrightarrow \beta + \beta' \longrightarrow \beta + \beta' + \alpha \longrightarrow \beta + \alpha$$

Often very long times (>50 hours) are necessary to complete the transformation and precipitation of alpha phase.

1.4 Cold Work:

Cold work is defined as deforming a metal plastically at a temperature below its recrystallization temperature. Cold working of a metal results in an increase in strength or hardness and decrease in ductility. As metals are deformed, the dislocation density increases and the energy in the system also increases. These effects accelerate the rate of aging. The effect of cold working prior to aging is shown schematically is shown in Fig. 7. Cold working of an alloy prior to aging results in higher hardness or strength while maintaining good ductility.

CHAPTER 2

EXPERIMENTAL PROCEDURE

2.1 Sample Preparation:

The Beta-C titanium alloy bar which was 1.250 inch in diameter was cut into 0.25 inch thick samples. The alloy was solution heat treated at 816°C (1500°F) for 1 hour and air cooled. Beta-C titanium alloy bars were solution heat treated at 816°C (1500°F) for 1 hour and air cooled prior to cold working 14.7%, 28.3%, and 39.1%. These cold work bars were also cut into 0.25 inch thick samples. These bars were supplied by RMI company.

2.2 Aging of Solution Treated Samples:

The solution heat treated samples were wet ground prior to the hardness measurement. The hardness was measured using the Rockwell hardness tester on 'C' scale. Two sets of samples were made to carry out the aging treatment at two different temperatures. One set of samples was aged at constant temperature of 538°C (1000°F) for 1, 2, 3, 4, 5, and 20 hours and air cooled. The second set of samples was aged at constant temperature of 427°C (800°F) for 1, 2, 3, 4, 5, 7, and 20 hours and air cooled. Hardness data were measured for both sets of samples on Rockwell hardness tester after aging. Optical microscopic examination and micro-Vickers hardness tests were performed on specific samples.

2.3 Aging of Cold Work Samples:

Each cold worked sample was wet ground on one side to measure hardness and the other side of the each sample was left as it was after cutting. The hardness was measured using the Rockwell hardness tester on 'C' scale. Two sets of samples were made. Each set consists of 14.7%, 28.3% and 39.1% cold work samples. These two sets were aged at the same temperatures (538°C and 427°C) and times as in the case of unworked samples. Hardness data were measured for both sets of samples on Rockwell hardness tester. After the aging treatment, the second sides of the samples were ground for both sets and hardness data were measured. Optical microscopic examinations were performed on specific samples.

2.4 Aging for Long Times:

The unworked samples which were aged at 427°C and 538°C were renormalized at 816°C (1500°F) for 1 hour and air cooled. The hardness data were measured after renormalizing. These samples were aged at constant temperature of 427°C (800°F) with time ranging from 1 to 20 days and then air cooled. Hardness data were measured on Rockwell hardness tester after the aging treatment.

2.5 Aging of Renormalized Samples:

The unworked and cold worked samples which were aged at 538°C (1000°F) and 420°C (800°F) were renormalized at 816°C (1500°F) for one hour and air cooled. The hardness data were measured after renormalizing. These samples were aged again at the same temperatures (538°C and 427°C) and times as the first time and air cooled. Hardness data were measured on a Rockwell hardness tester.

2.6 Mechanical Tests:

Rockwell hardness tests of Beta-C titanium samples were made by using the 'C' scale. The 'C' scale uses a major load of 150-Kg and Diamond cone as an indenter. Micro-Vickers hardness tests of a Beta-C Ti specimen were made on a Leit-Wetzlar mini load hardness tester at a load of 200 grams.

2.7 Optical Microscopy:

Optical microscopy was carried out to determine the microstructural changes after aging treatment at different temperatures and times. The specimens were cross-sectionally cut in the centre and mounted using an automatic metallographic mounting press. The specimens were wet ground by SiC paper with grits 60, 80, 120, 320, 800, 1200, polished by Diamond powder down to 1 μm and then etched in 2 HF, 1 HNO_3, 17 H_2O, solution. Specimen were examined using standard optical techniques, and photographs of the samples were taken on Zeiss-Axiophot Metallograph using polaroid type 53 and 52 film.

CHAPTER 3

RESULTS

3.1 Aging Curves for Solution Treated Alloy:

Figs. 8 through 10 show the effect of aging time at 538°C (1000°F) and 427°C (800°F) on the hardness of Beta C alloy. The Fig. 8 shows the effect of aging time at 538°C (1000°F) on the hardness of the alloy. The alloy shows maximum hardness of 41 HRC after aging for 4 hours. Fig. 9 shows the effect of aging time at 427°C (800°F) on the hardness of the alloy. The alloy shows maximum hardness, which is 39 HRC after aging for 7 hours. In Fig. 10, both curves are drawn for comparing the effect of aging temperature and time on the hardening of alloy. Fig. 11 shows the effect of long aging time at 427°C (800°F) on the hardness of the alloy. The curve shows that the hardness remains at a maximum value on aging from 10 to 14 days. This number is about 50 HRC. Fig. 12 shows the Vicker hardness of the alloy specimen which had been aged at 427°C (800°F) for 7 hours and air cooled. This particular specimen was selected for Vicker hardness testing because it has dark areas on edges. The microhardness data were measured by going across the specimen, i.e., from one edge of specimen to other edge. Hardness versus distance across the specimen is plotted in Fig. 12. This curve has a U shape which shows higher hardness on the edges and lower hardness in the center i.e., away from the edges. The hardness number is also very high in dark areas of the specimen. Fig. 12 also shows equivalent HRC numbers for the Vicker hardness numbers.

3.2 Aging Curves for Cold-Worked Alloy:

Figs. 13 through 17 show the effects of different amounts of cold working and aging times at 538°C (1000°F) on the hardness of Beta-C. Fig. 13 shows the effect of aging time on the hardness of the 14.7% cold worked alloy. This alloy shows maximum hardness of 44 HRC after aging for 3 hours. Fig. 14 shows the effect of aging time on the hardness of the 28.3% cold worked alloy. This alloy shows maximum hardness of 45 HRC after aging for 2 hours. Fig. 15 shows the effect of aging time on the hardness of the 39.1% cold worked alloy. This alloy shows maximum hardness of 45 HRC after aging for 1 hour. In Fig. 16, all three curves for 14.7% , 28.3% and 39.1% cold work are plotted together to see the hardness profiles. In Fig. 17, the hardness-aging time curves are plotted after grinding the second sides of the cold work and aged specimens.

The hardness values after grinding the second sides of aged specimens are almost the same as for the first sides after aging. Fig. 16 and 17 reveal that the specimens are not hardened by oxygen contamination during aging. This hardening is due to the precipitation of α in beta matrix.

Figs. 18 through 22 show the effect of aging time at 427°C (800°F) on the hardness of 14.7%, 28.3% and 39.1% cold work alloy. Fig. 18 shows the effect of aging time at 427°C (800°F) on the hardness of 14.7% cold worked alloy. This alloy shows maximum hardness of 47 HRC after aging for 7 hours. Fig. 19 shows the effect of aging time at 427°C (800°F) on the hardness of 28.3% cold work alloy. This alloy shows maximum hardness of 49 HRC after aging for 7 hours. Fig. 20 shows the effect of aging time at 427°C (800°F) on the hardness of 39.1% cold work alloy. This alloy shows maximum hardness of 50 HRC after aging for 20 hours. In Fig. 21, all three curves for 14.7%, 28.3% and 39.1% cold work are plotted together to analyze the hardening of alloy. In Fig. 22, the hardness-aging time curves are plotted after grinding the second side of the cold worked and aged specimens. The hardness values after grinding the second side of aged specimens are almost the same as for the first sides after aging. Fig. 21 and 22 reveal that specimens are not hardened by oxygen contamination during the aging. This increase in hardness is due to precipitation of α in beta matrix.

3.3 Aging Results of Renormalized Samples:

The unworked and cold worked samples did not show increase in hardness after aging treatment. The hardness almost remains the same as it was in the normalized condition in both cases. This behavior is unusual for the unworked Beta-C alloy and further study is required to investigate the aging behavior of the alloy. In the case of cold work, the alloy did not show increase in hardness. The hardness remains the same as it was in the normalized condition. A possible explanation for this effect is that the renormalization treatment negated the internal strain due to the cold work, thus, eliminating nucleation sites for age hardening.

3.4 Microstructures study:

Figs. 23 through 25 show the microstructures of solution-treated and aged Beta-C alloy. In Fig. 23, the specimen was solution treated at 816°C (1500°F) for 1 hour and air cooled. Fig. 23 shows secondary phases that may have developed in the beta matrix from trace impurities. Fig. 24 shows the microstructure of an alloy which was solution treated at 816°C (1500°F) for 1 hour, air cooled and aged at 427°C (800°F) for 1 hour and air cooled. Alpha precipitates are not visible in the beta matrix in Fig. 24 due to their small size and short aging time. Fig. 25 shows the microstructure of an alloy which was solution treated at 816°C (1500°F) for 1 hour, air cooled and aged at 427°C (800°F) for 7 hours and air cooled. The large dark areas on the bottom side are more heavily aged, with a greater amount of α phase, and are harder than the lighter, all beta grains. The darker areas appear to have been contaminated by oxygen during solution treating. Oxygen is a strong α stabilizer and accelerates the aging reaction. Figs. 26 through 28 show the microstructures of cold work and aged Beta-C alloy. Fig. 26 shows the microstructure of an alloy which was solution treated at 816°C (1500°F) for 1 hour, air cooled and then cold worked to 39.1%. It is obvious from the micrograph that grains in the beta matrix are elongated due to cold work. Fig. 27 shows the microstructure of the alloy, which was solution treated at 816°C for 1 hour, air cooled, 39.1% cold worked and aged at 538°C (1000°F) for 20 hours and air cooled. The alpha precipitates are very fine and can hardly be seen in the beta matrix in Figure 27. Fig. 28 shows the microstructure of an alloy which was solution treated at 816°C (1500°F) for 1 hour, air cooled, 39.1% cold worked and aged at 427°C (800°F) for 20 hours. The alpha precipitates are very fine and cannot be resolved but the specimen has a hardness of 50 HRC due to the precipitation of aging particles (alpha) in the beta matrix.

CHAPTER 4

DISCUSSION

Age hardening of an alloy depends on both temperature and the time for aging. Fig. 10 exhibits that the curves are quite temperature dependent. At an aging temperature of 538°C (1000°F) the diffusion in the Beta-C alloy is rapid and the α precipitates form quickly. Strength reaches a maximum after 4 hours whereas at a lower aging temperature of 427°C (800°F), longer times are required to produce the optimum strength. The long time aging treatment of alloy specimens at 427°C (800°F) resulted, as expected, in a significant increase in hardness in Fig. 11. A maximum increase in hardness was found after 10 days. The hardness almost remains maximum up to 14 days, then the curve shows a decrease in hardness and the alloy is overaged. A possible explanation for this phenomenon is that the α precipitates were small in size and aligned with the beta matrix, giving coherent precipitates. The hardness stayed at a maximum for the alloy in the aged condition. However, when the precipitates coarsen, mismatch occurs between the precipitates and the matrix, giving an incoherent precipitate. The hardness decreases in this overaged condition. The U shape curve in Fig. 12 shows higher hardness on the edges because of the presence of α particles. These particles are the large dark areas on the microstructure in Fig.25. This U-curve shows considerable increase in hardness at some points on the curve and this is due to the presence of α precipitates in the beta matrix. The Vickers hardness number drops whenever the indenter falls on the ductile beta matrix. The microstructure also shows dark parallel lines which are due to α precipitation on the slip planes in the beta matrix.

Judging from the aging curves in Fig. 13 to 22 the cold work promoted considerable hardening of the alloy as compared to the solution treated and aged alloy. As the amount of cold work increases, the hardening potential of alloy also increases but the curves are still quite temperature dependent. At a higher aging temperature of 538°C (1000°F), 39.1% cold work shows higher hardening potential as compared to the 14.7% and 28.3% cold working. As the amount of cold work increases, the aging time to reach a maximum hardness decreases in all curves (Figs.13-17). The 39.1% cold worked alloy took only 1 hour to reach a maximum hardness whereas in the case of 14.7% and 28.3% cold work, the alloys took comparatively longer times to attain maximum strength. After reaching the maximum hardness, all three amounts of cold work show only a small decrease in hardness as the alloy is overaged. At a lower aging temperature of 427°C (800°F), the 39.1% cold worked alloy also shows higher hardening potential as compared to 14.7% and 28.3% cold work. As the amount of cold work increases, the aging time to reach a high hardness level decreases as shown in curves (Figs.18-22). The 39.1% cold worked alloy took a shorter time to reach a high hardness level whereas in the case of 14.7% and 28.3% cold worked, the alloy took a comparatively longer time to reach a high strength level. After reaching the maximum hardness, the 14.7%, 28.3% and 39.1% cold worked alloy did not show any sign of overaging for up to 20 hours. The possible explanation for this greater aging response is that the cold worked alloy has a much greater number of dislocations. The more dislocation an alloy has, the more likely they are to interfere with one another, and the stronger the alloy becomes. This demonstrates that the aging after cold working can result in a higher hardness as α phase precipitates in the beta matrix.

CHAPTER 5

CONCLUSION

1. Aging after cold working shows higher hardening potential as compare to the simple aging.

2. The aging response for the cold worked Beta-C alloy at 538°C (1000°F) is very quick.

3. The aging response for the cold worked Beta-C alloy at 427°C (800°F) is relatively slow compared to aging at 538°C.

4. The alloy also shows higher hardness values at 427°C (800°F), as compared to 538°C (1000°F), with the same amount of cold work.

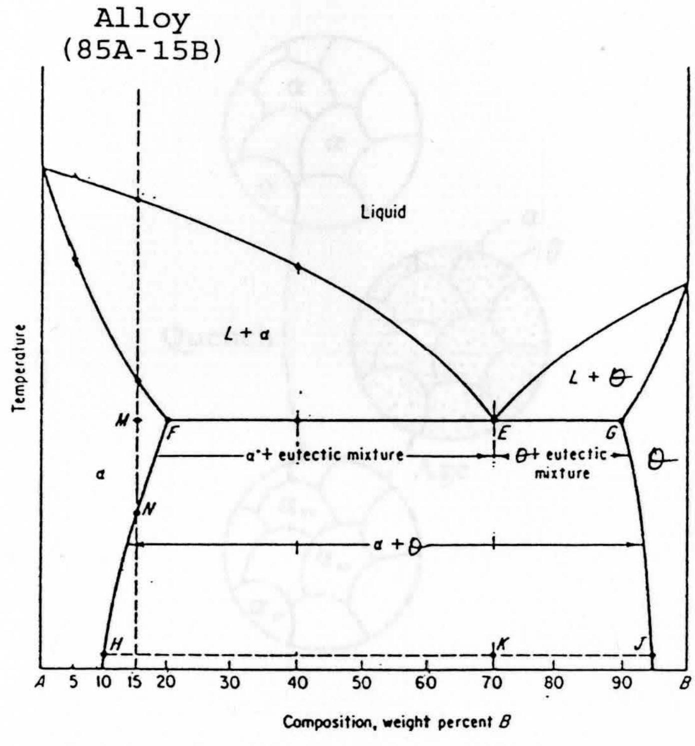


Fig. 1 Phase diagram illustrating partial solid solubility [10].

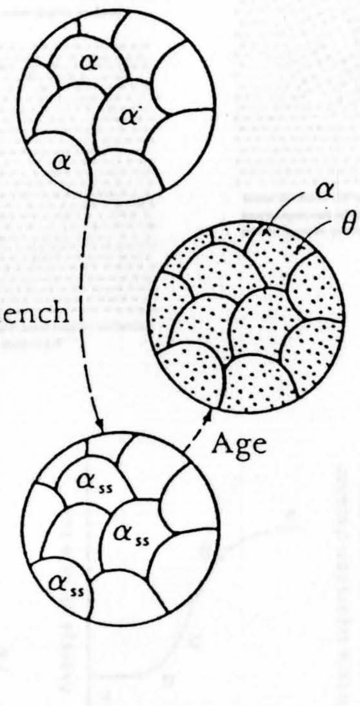


Fig. 2 Microstructures of age-hardening heat treatment [11].

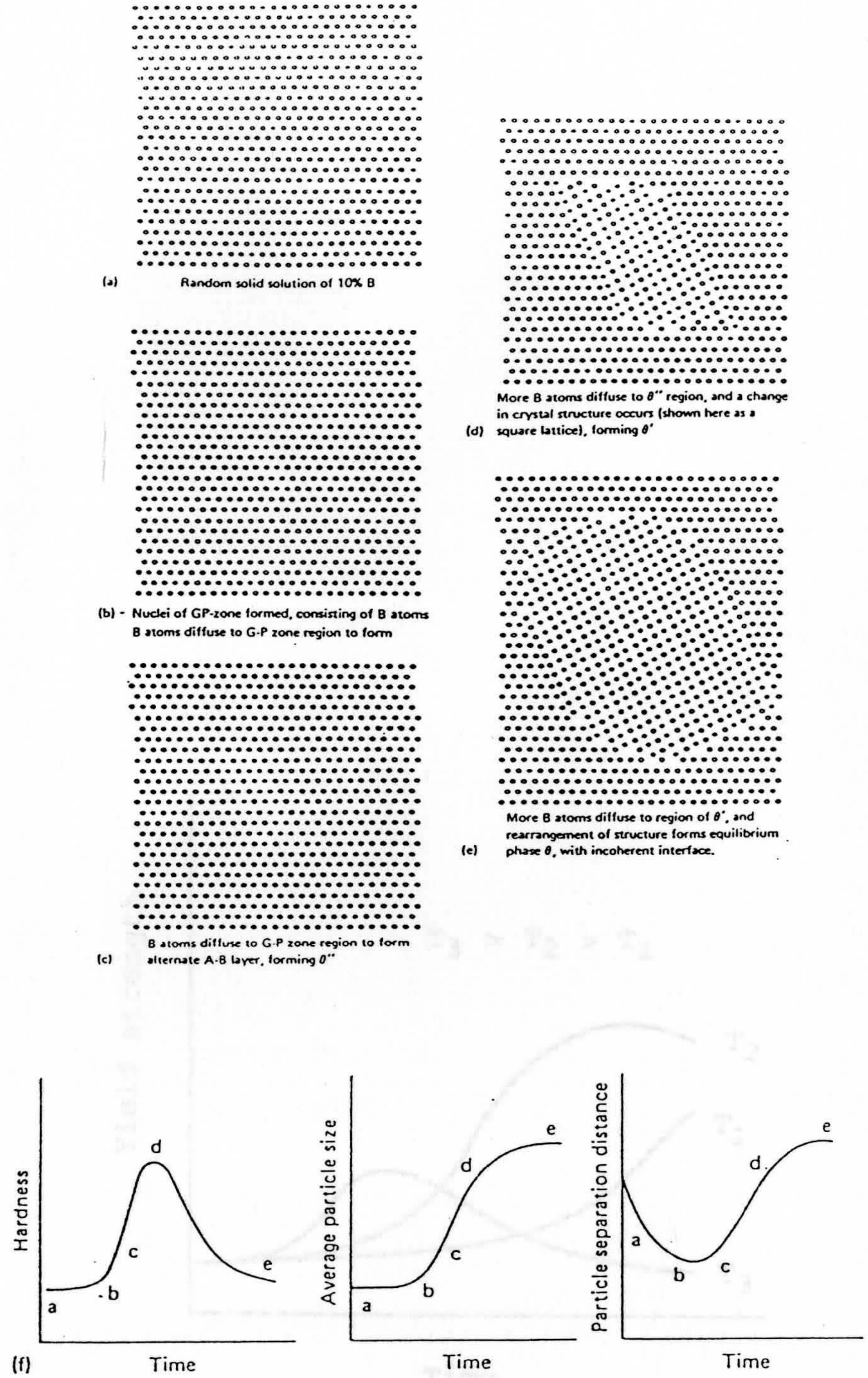


Fig. 3 Schematic illustration of a possible precipitation sequence [12].

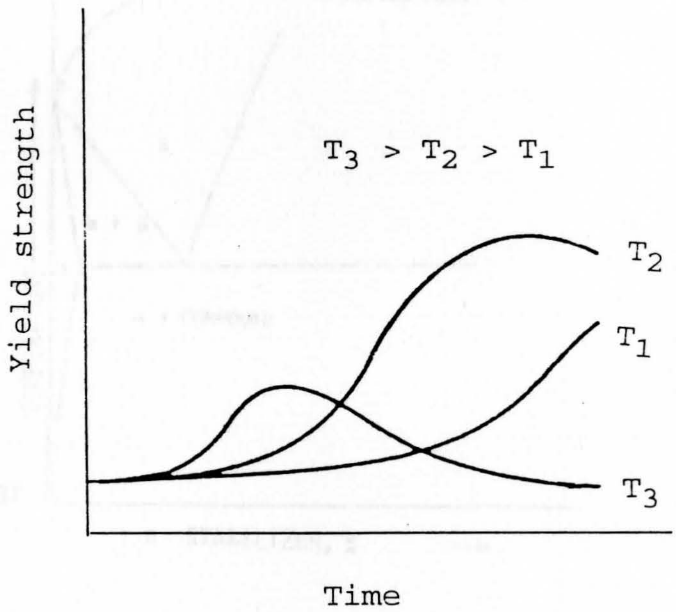
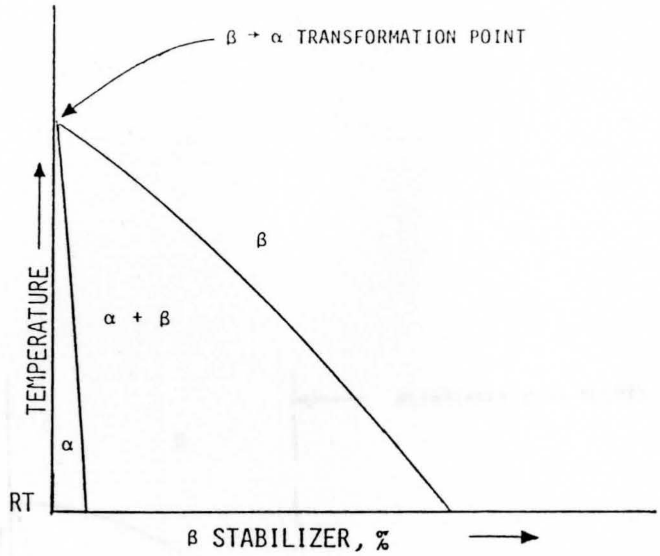
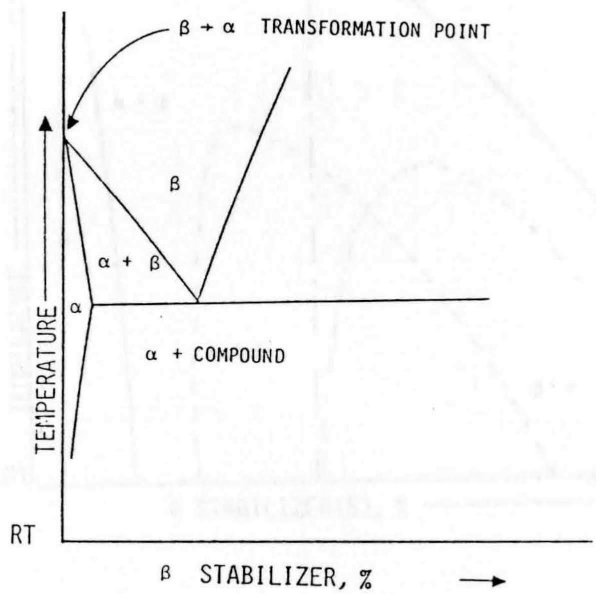


Fig. 4 Schematic variation of yield strength with aging temperature.



(a)



(b)

Fig. 5 Schematic phase diagrams for the two classes of beta stabilizers (a) beta isomorphous and (b) beta eutectoid [13].

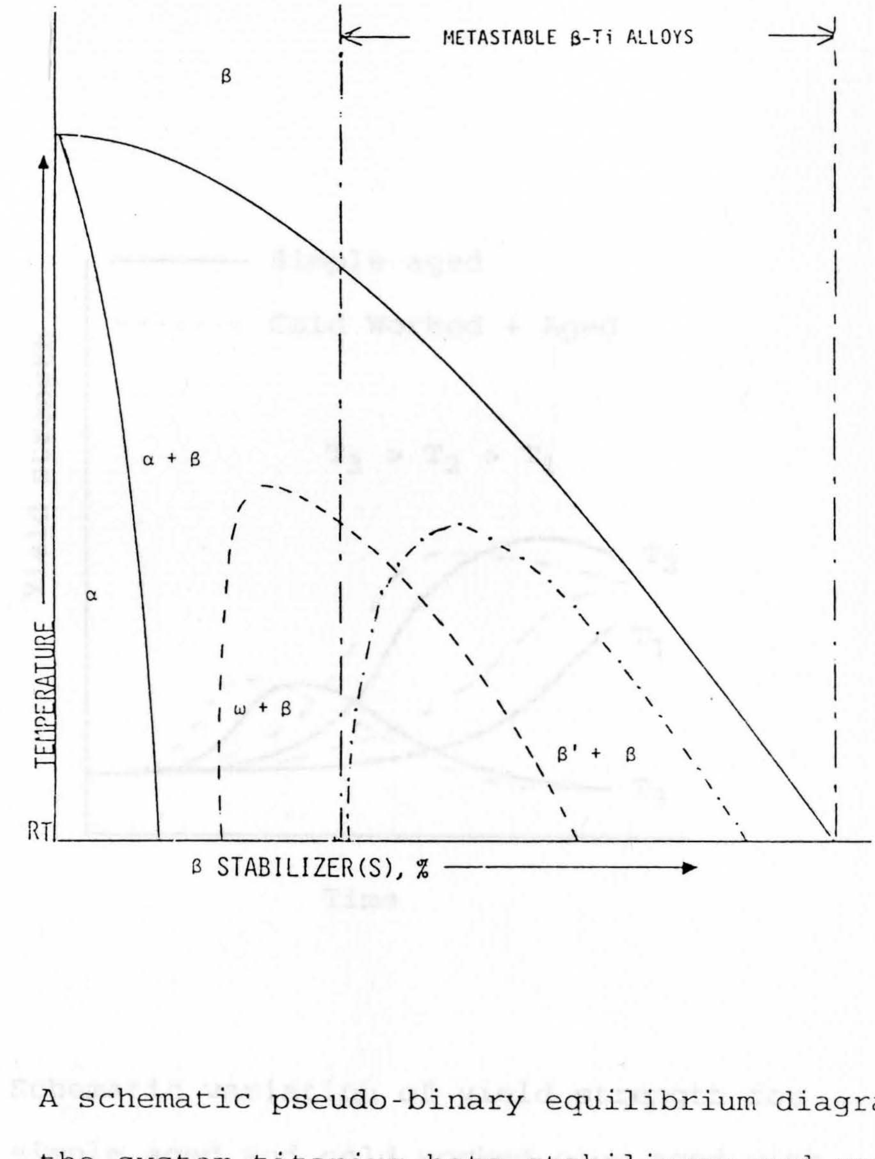


Fig. 6 A schematic pseudo-binary equilibrium diagram of the system titanium-beta stabilizers and ranges of omega and ß prime stability superimposed [14].

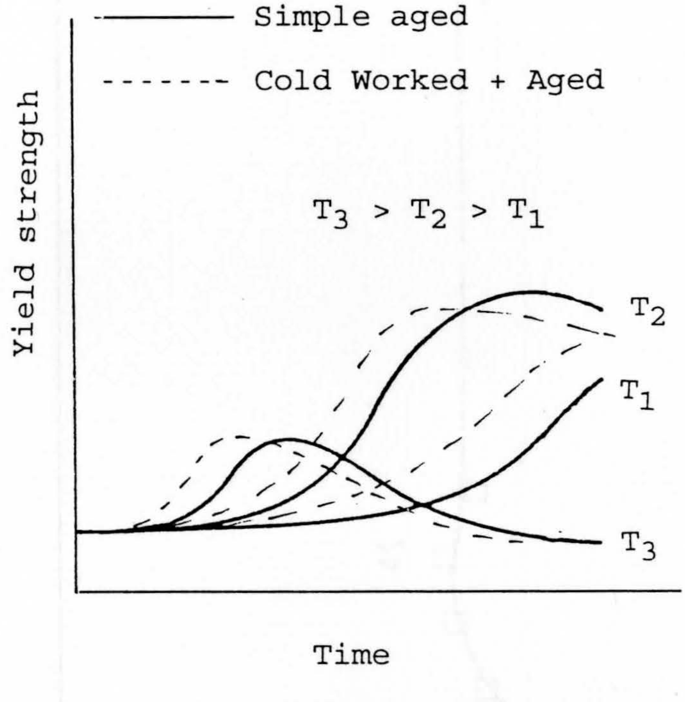


Fig. 7 Schematic variation of yield strength for simple aged and cold worked plus aged with aging temperature.

FIG.8 HARDNESS VS AGING TIME AT 538°C

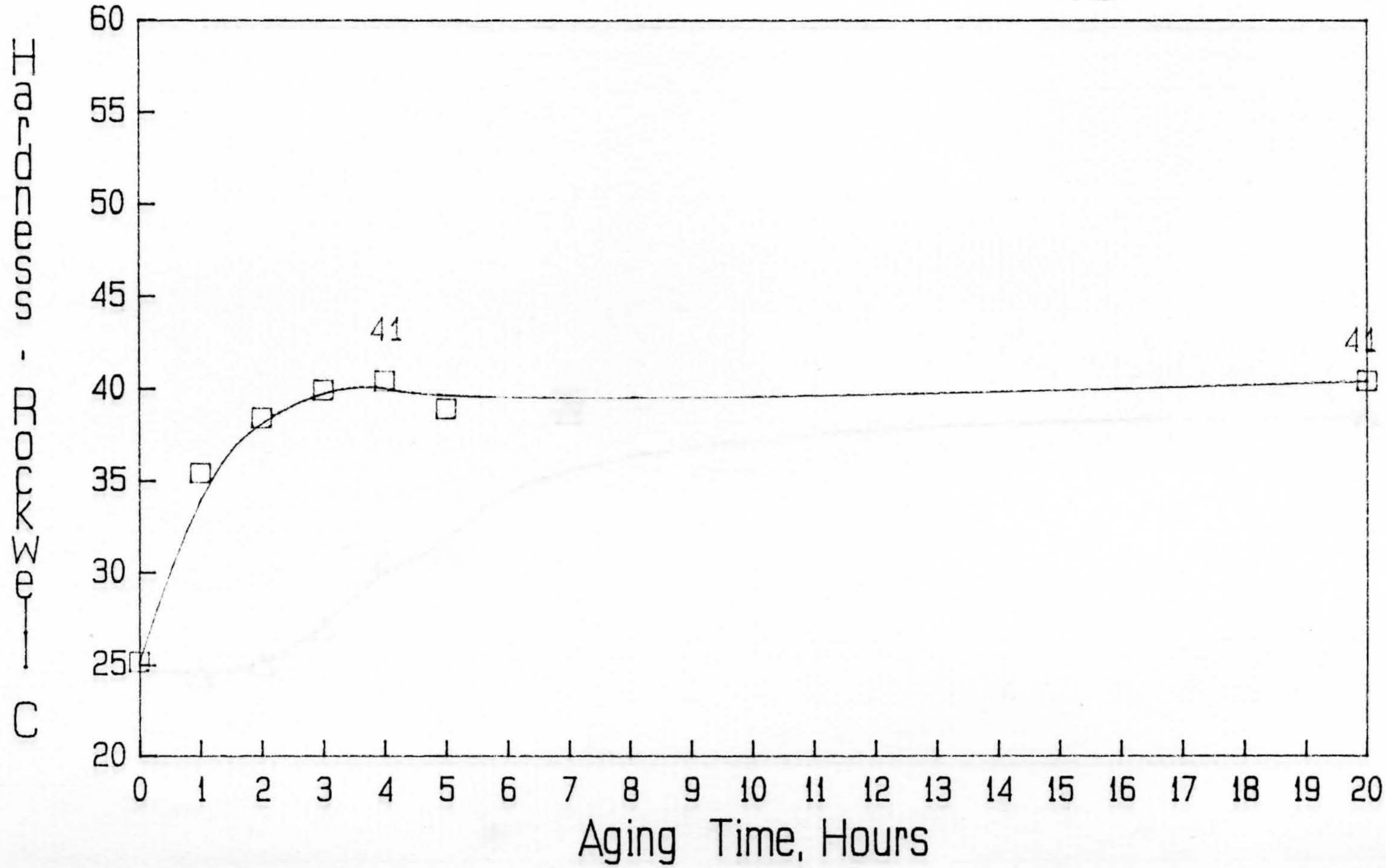


FIG.9 HARDNESS VS AGING TIME AT 427°C

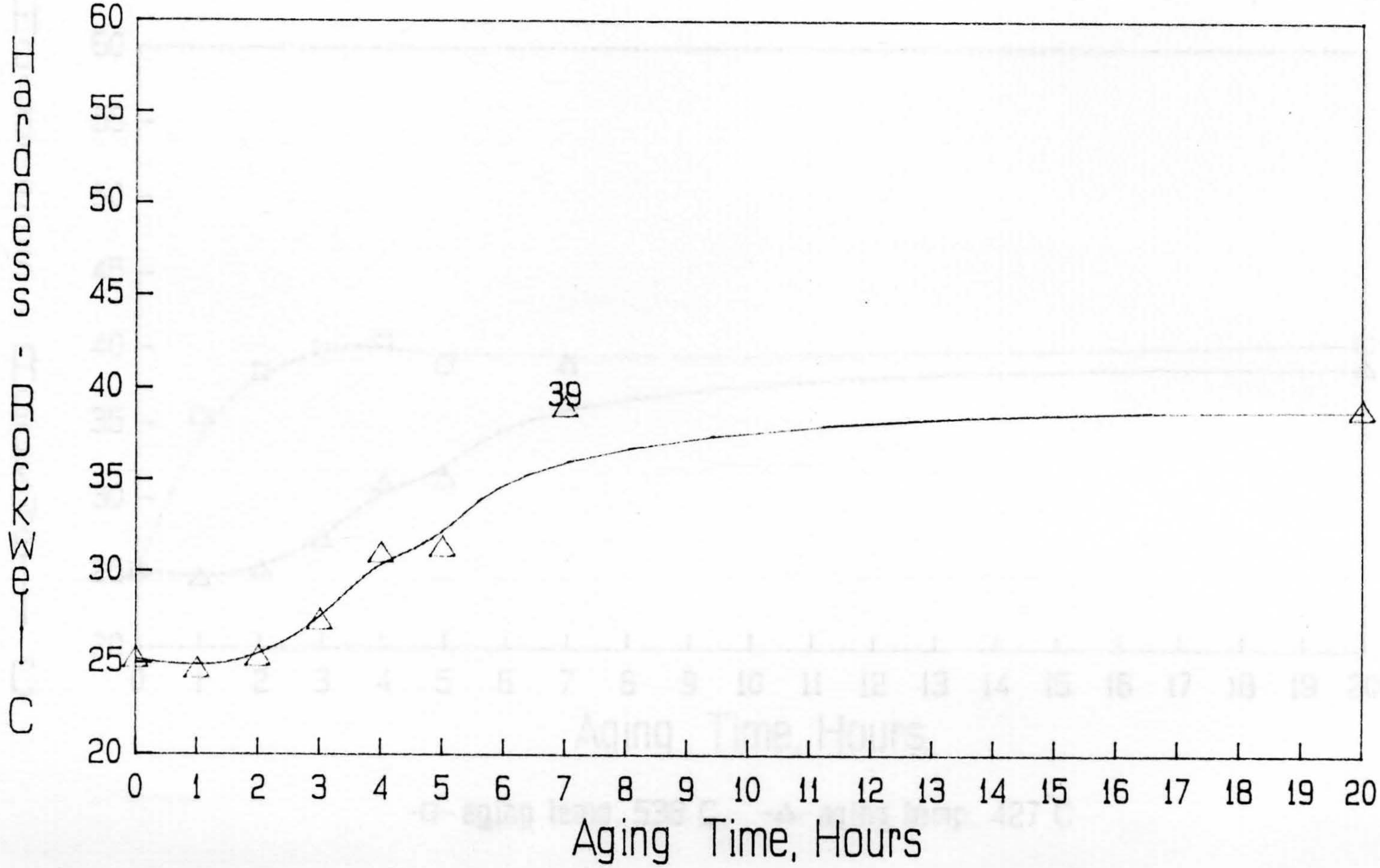


FIG.10 HARDNESS VS AGING TIME

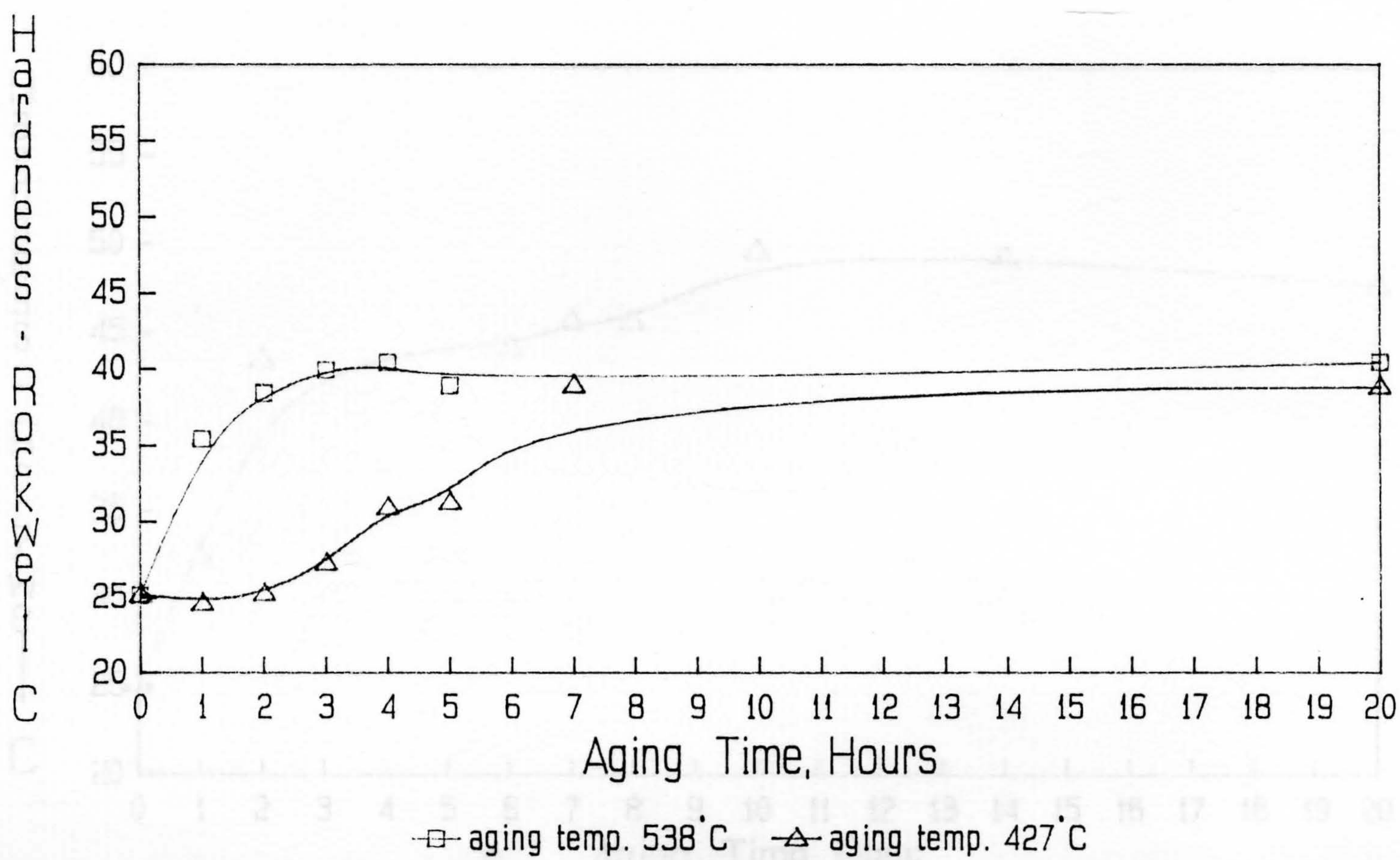


FIG.11 HARDNESS VS AGING TIME AT 427°C

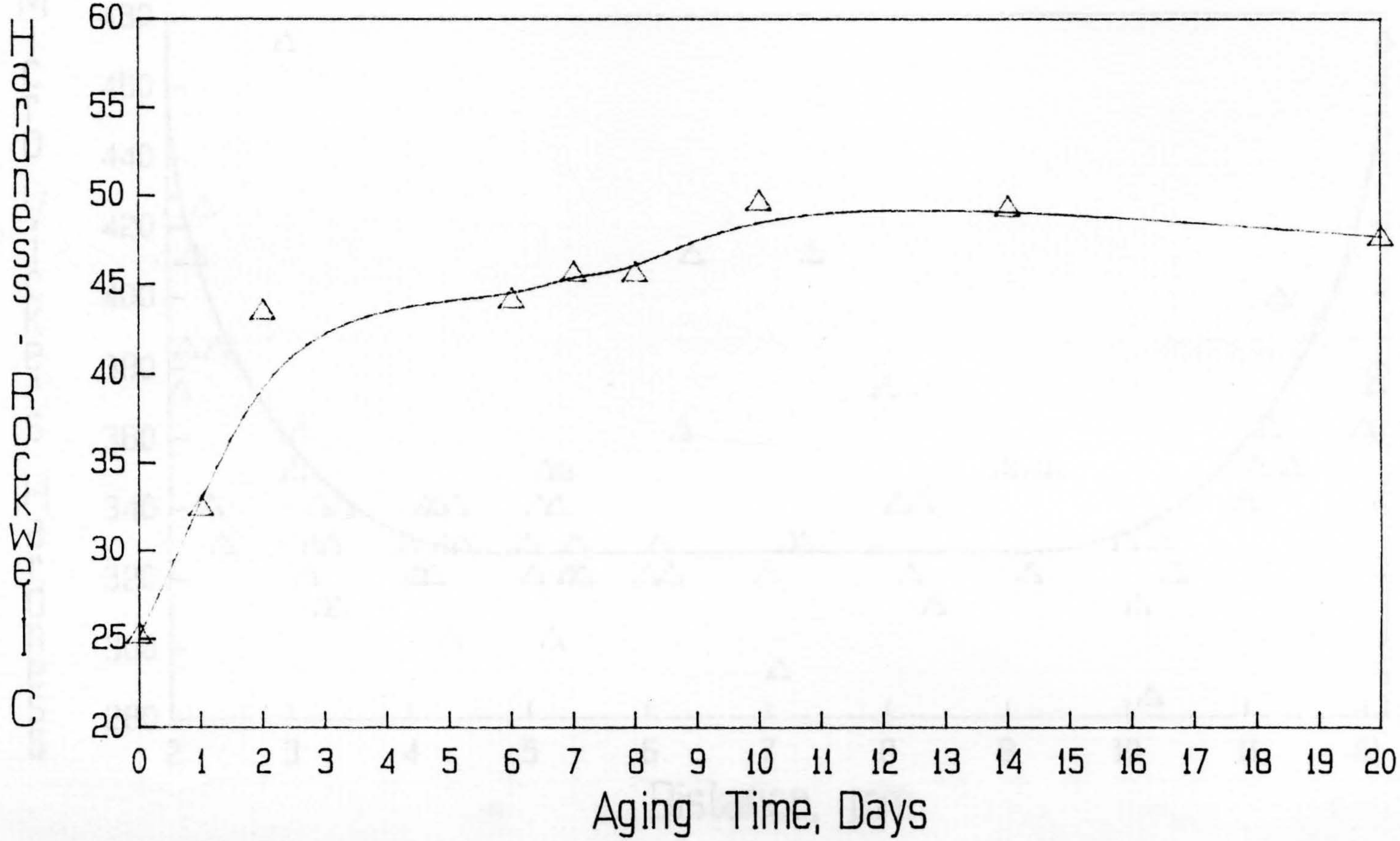


FIG.12 HARDNESS VS DISTANCE

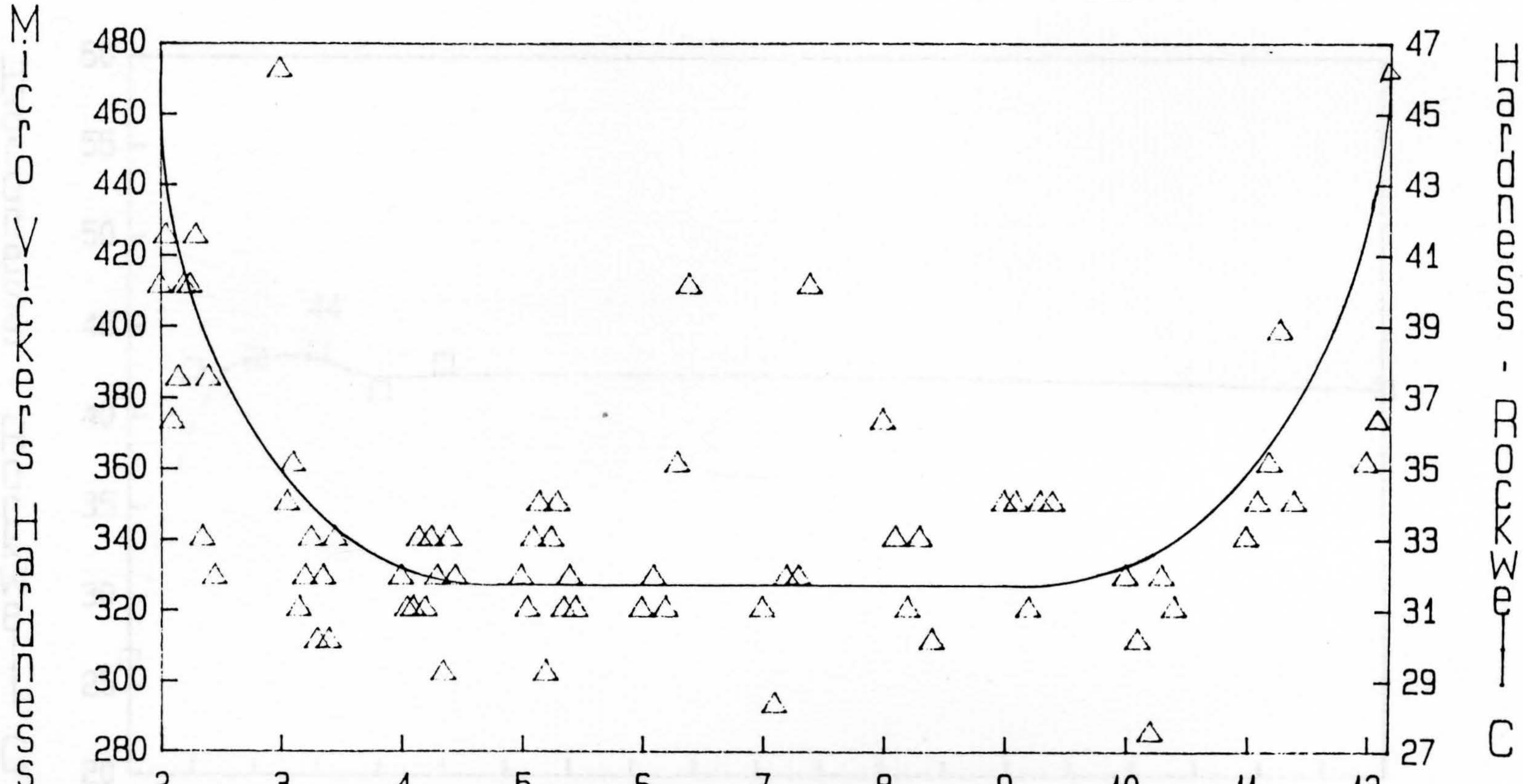


FIG.13 HARDNESS VS AGING TIME AT 538°C
FOR 14.7% COLD WORK

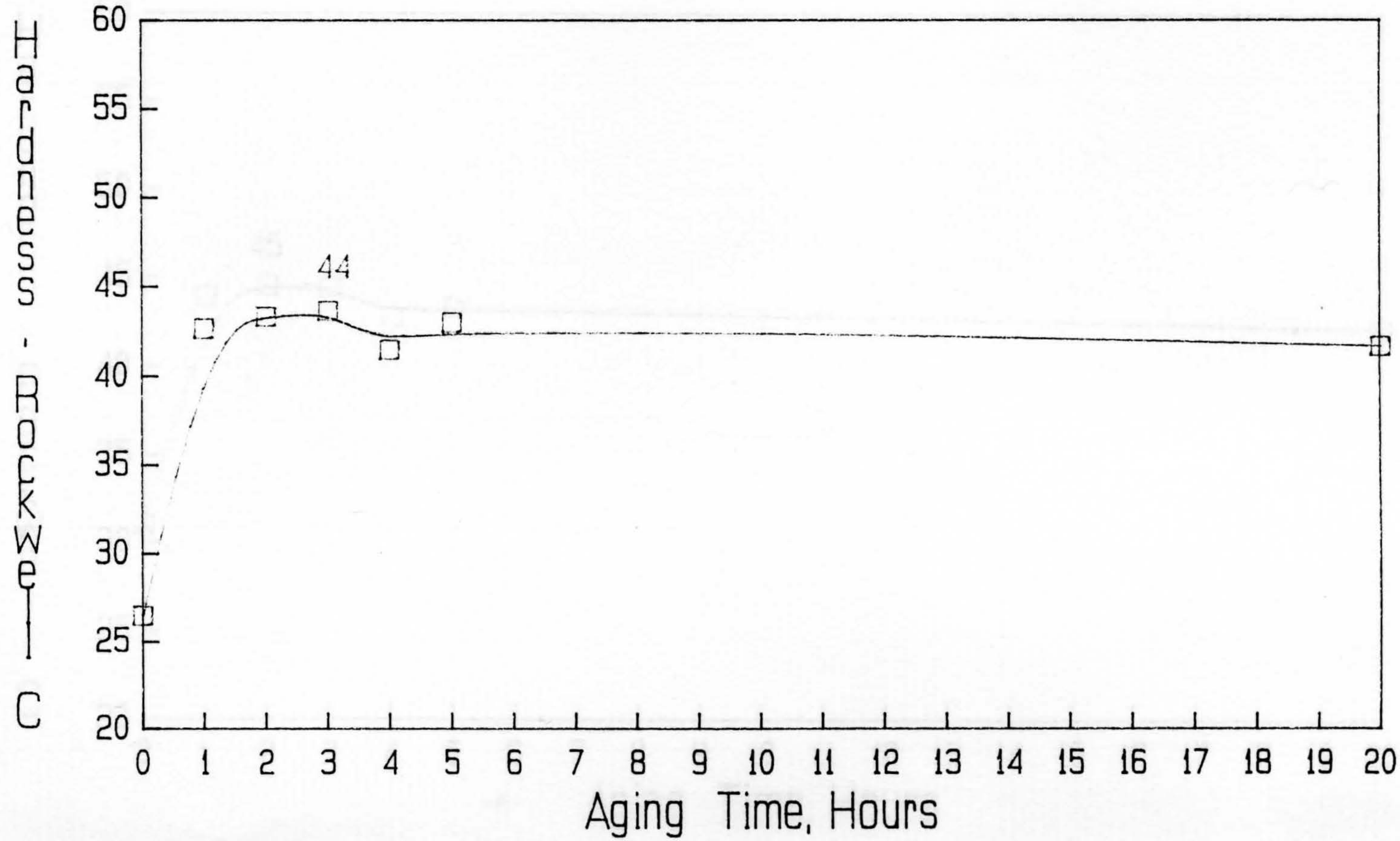


FIG.14 HARDNESS VS AGING TIME AT 538°C
FOR 28.3% COLD WORK

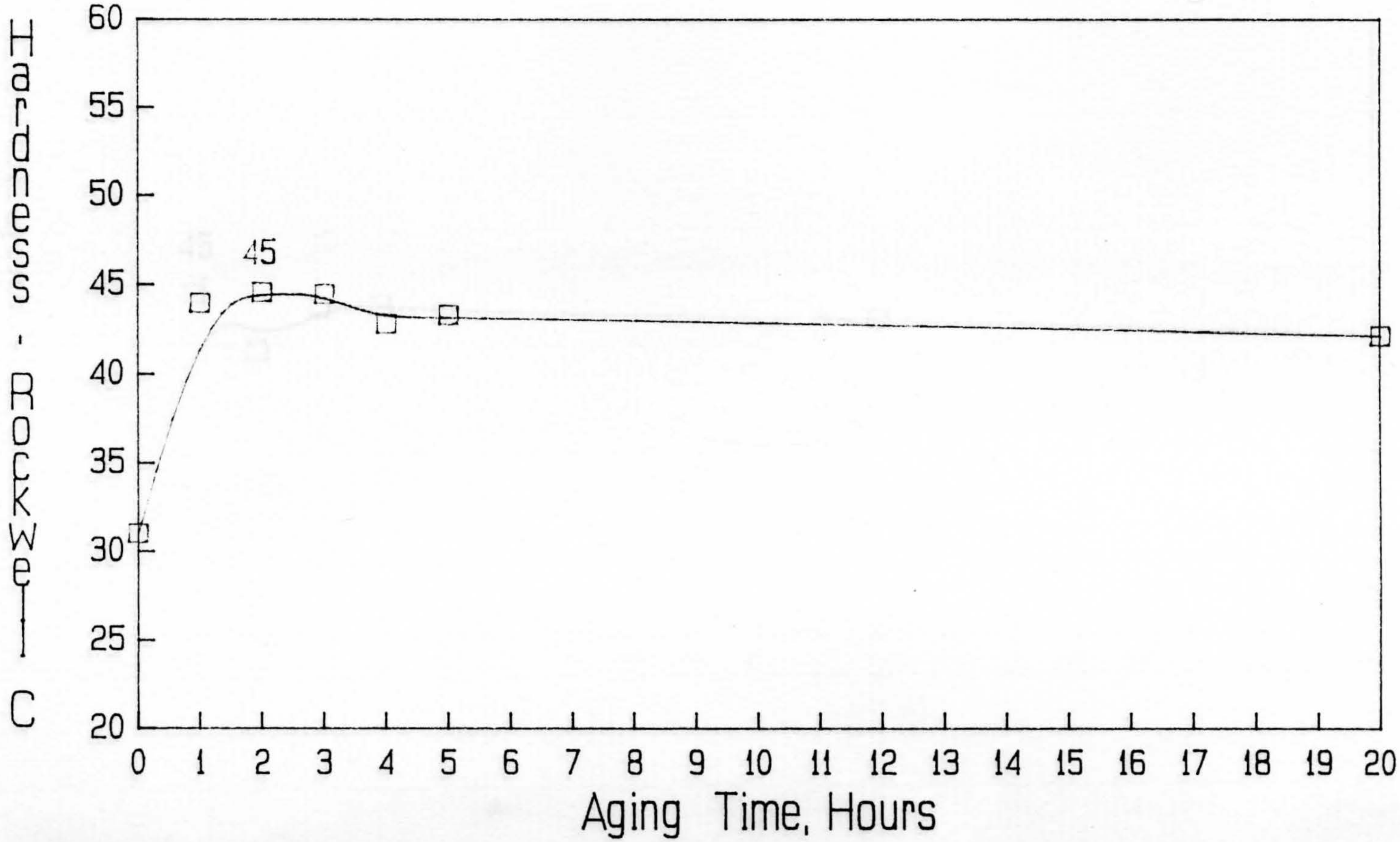


FIG.15 HARDNESS VS AGING TIME AT 538°C
FOR 39.1% COLD WORK

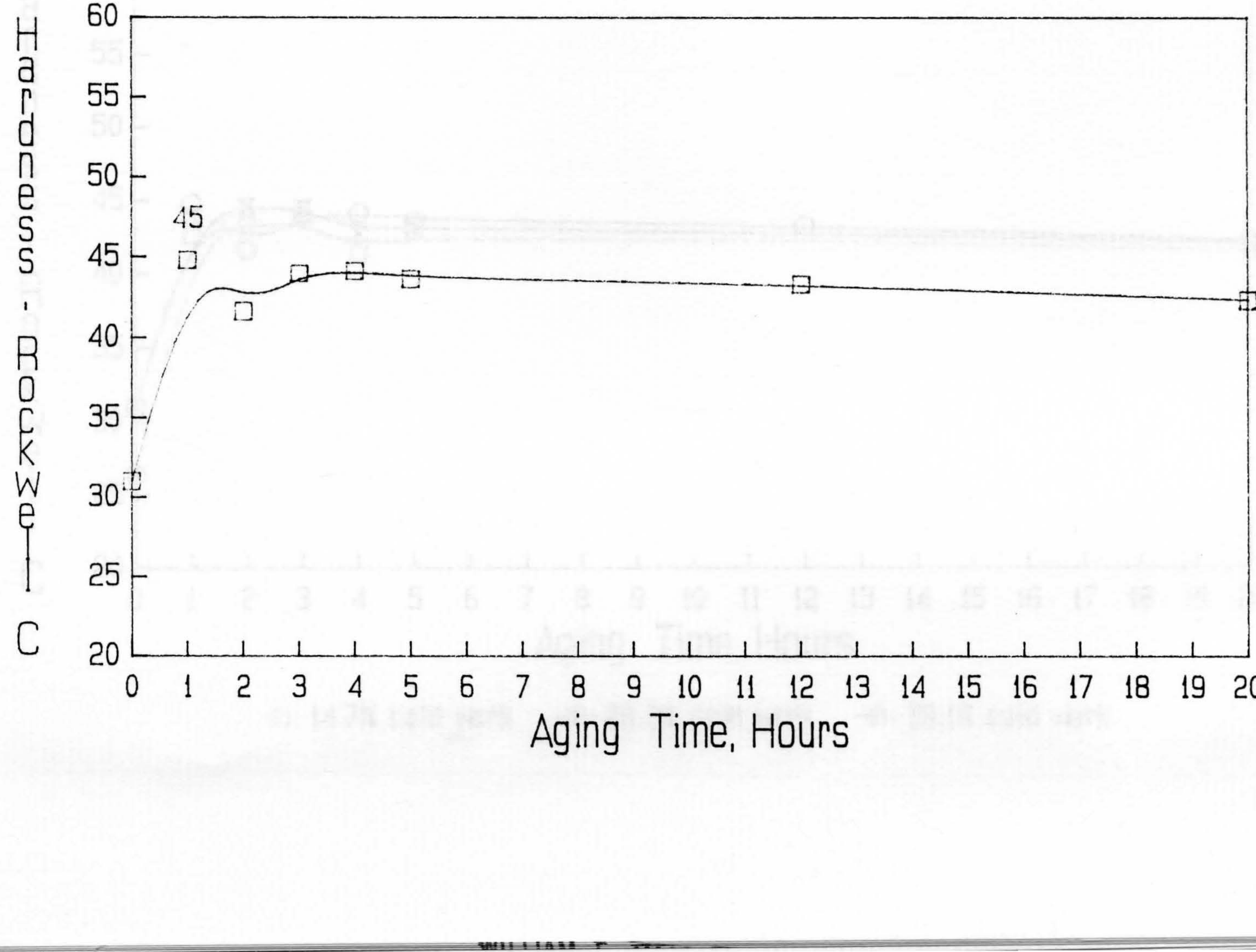


FIG.16 HARDNESS VS AGING TIME AT 538°C

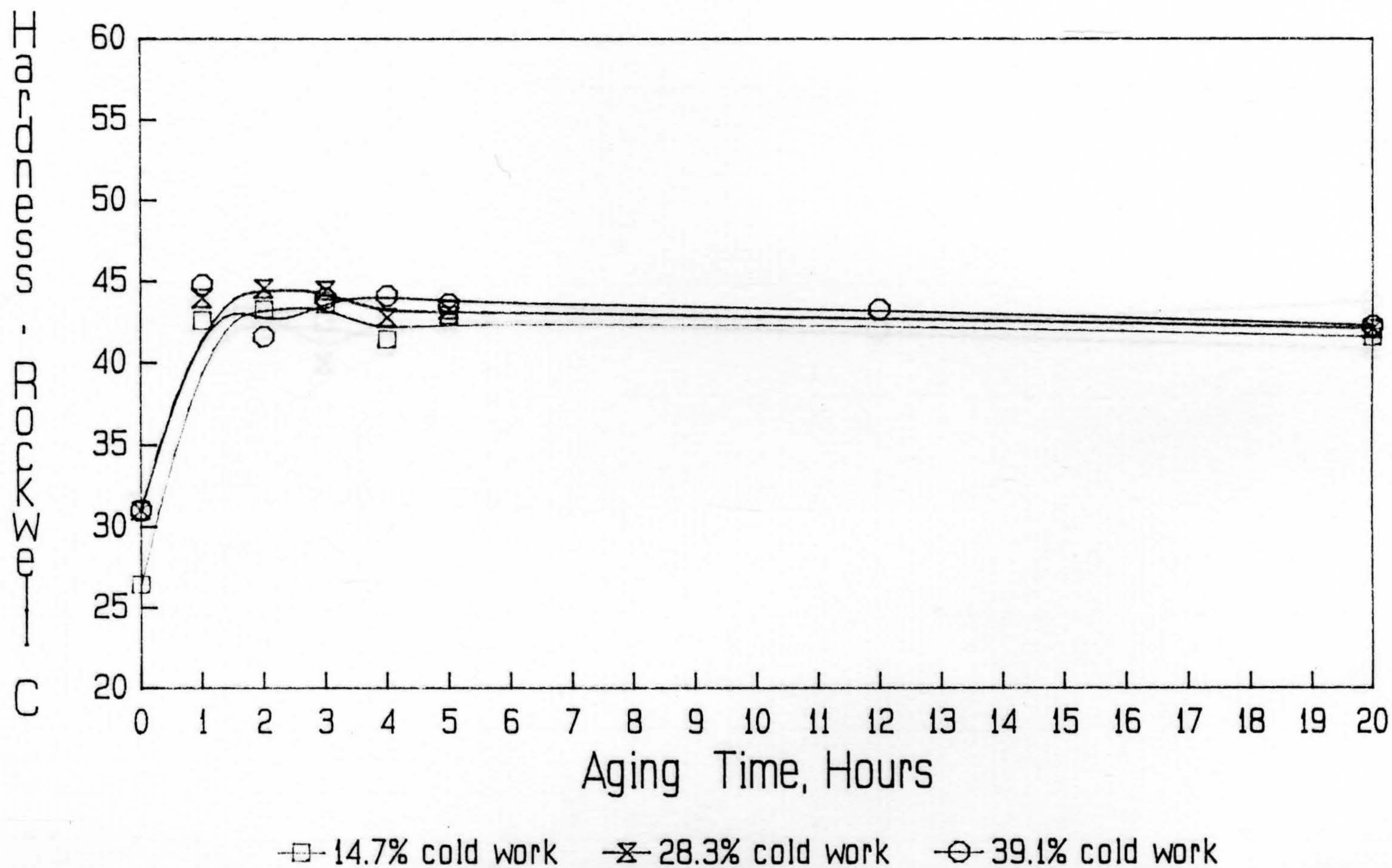


FIG.17 HARDNESS VS AGING TIME AT 538°C

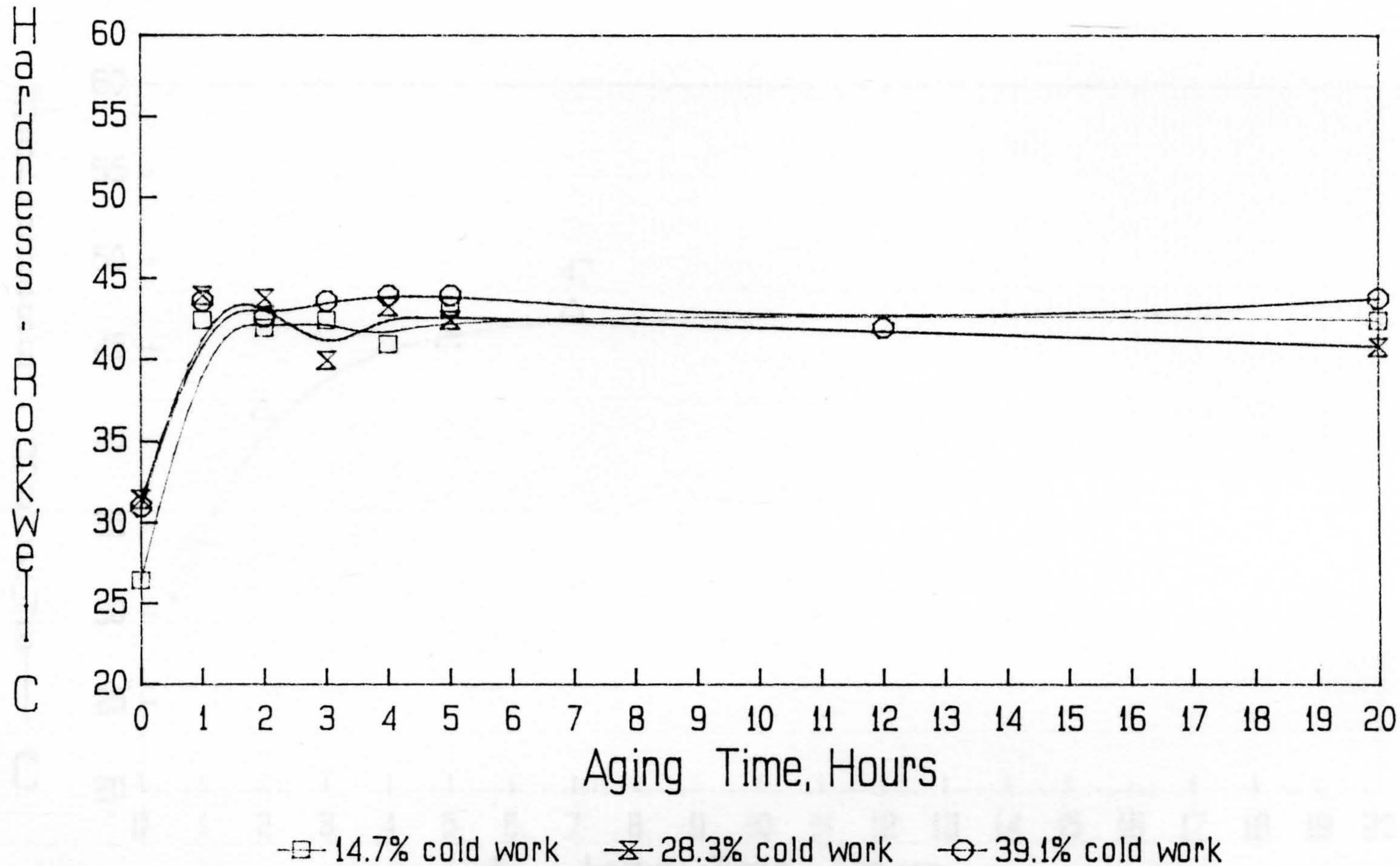


FIG.18 HARDNESS VS AGING TIME AT 427°C
FOR 14.7% COLD WORK

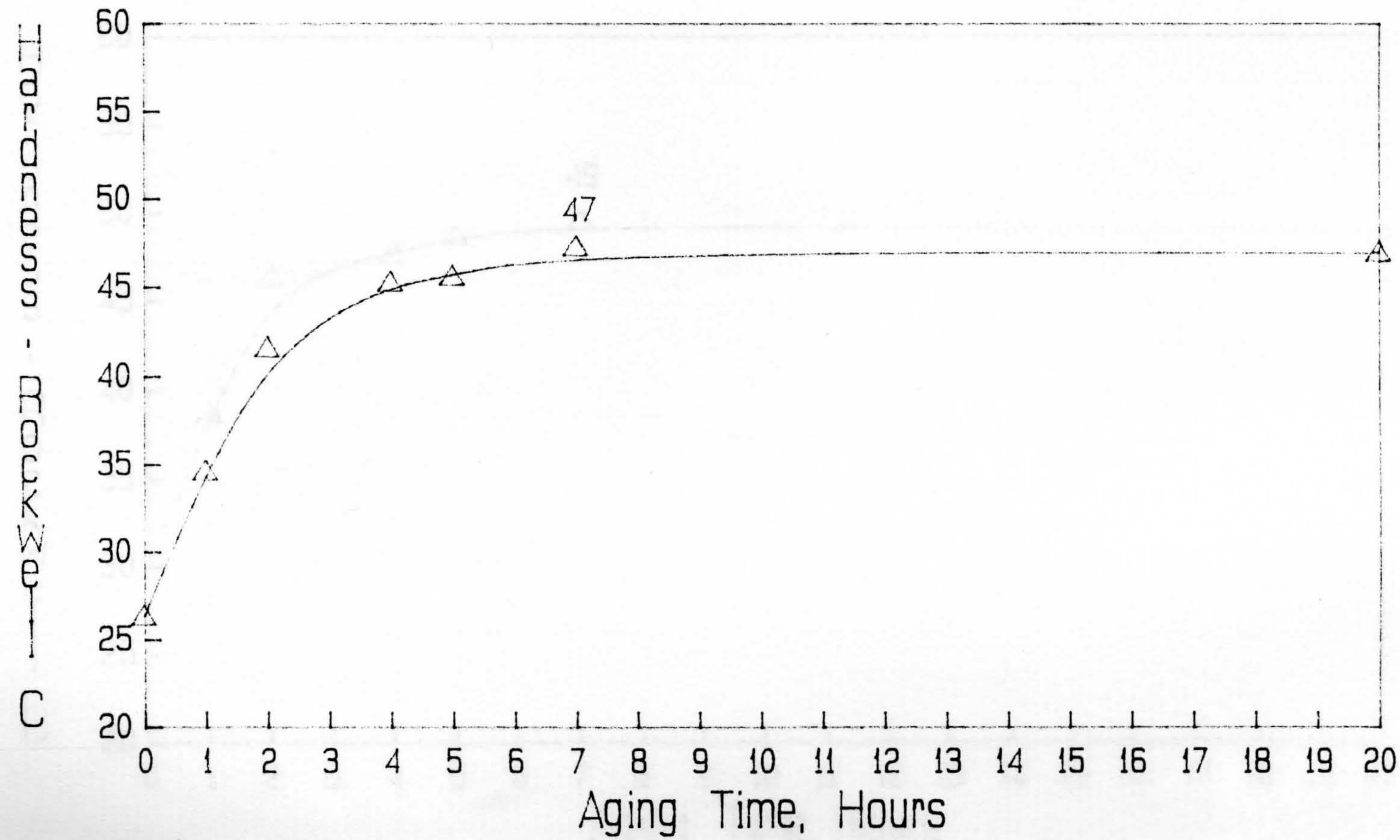


FIG.19 HARDNESS VS AGING TIME AT 427°C
FOR 28.3% COLD WORK

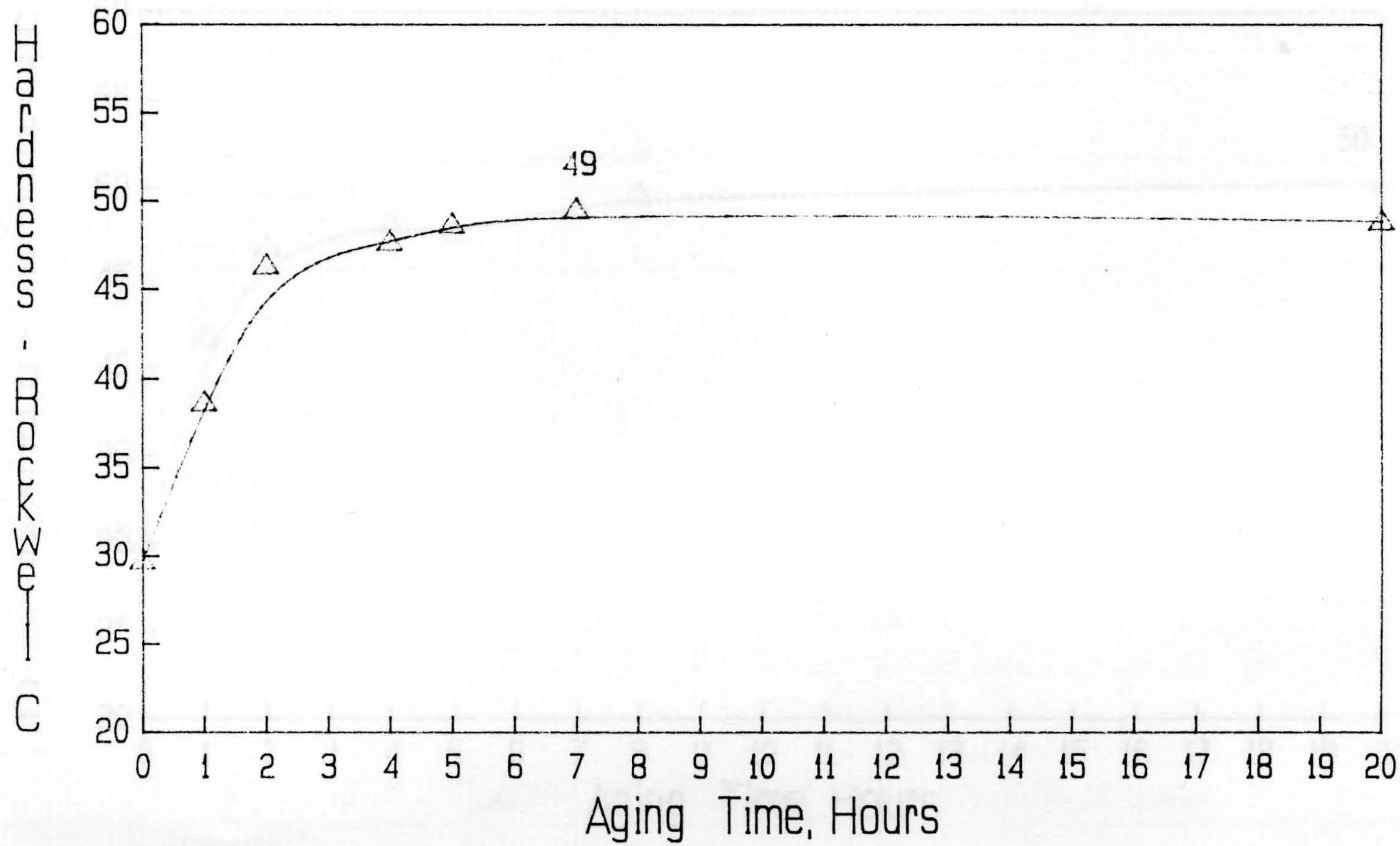


FIG.20 HARDNESS VS AGING TIME AT 427°C
FOR 39.1% COLD WORK

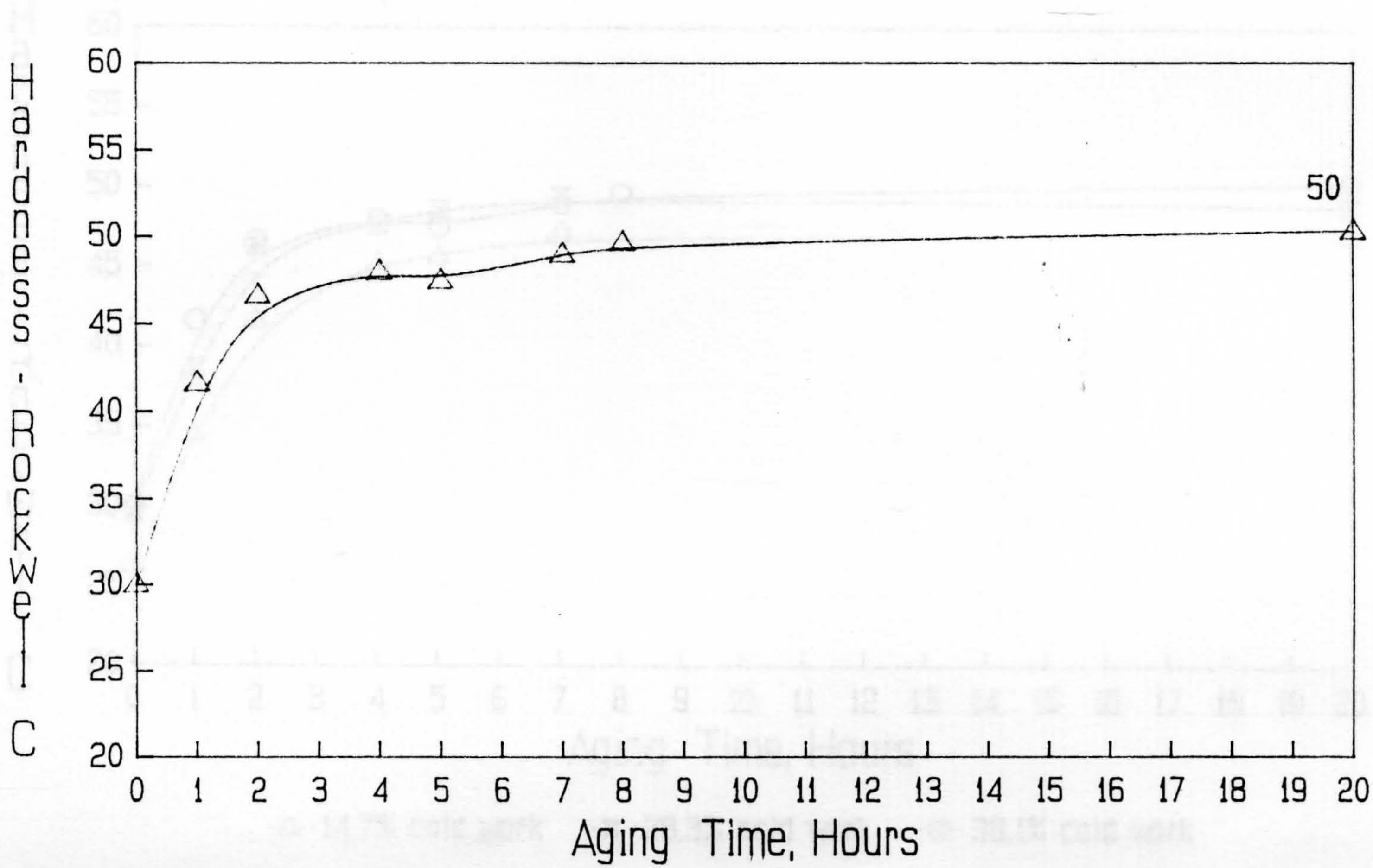


FIG.21 HARDNESS VS AGING TIME AT 427°C

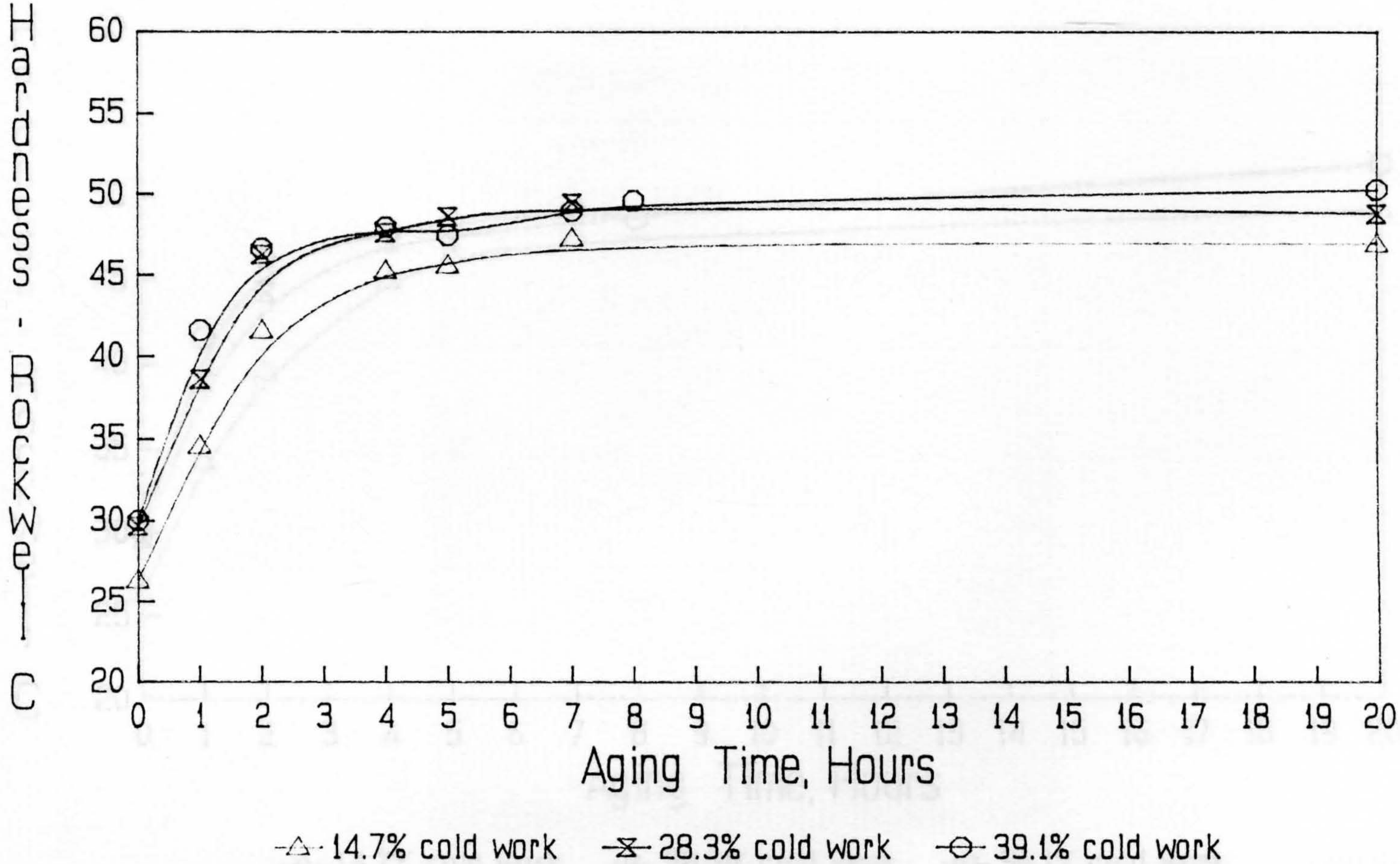
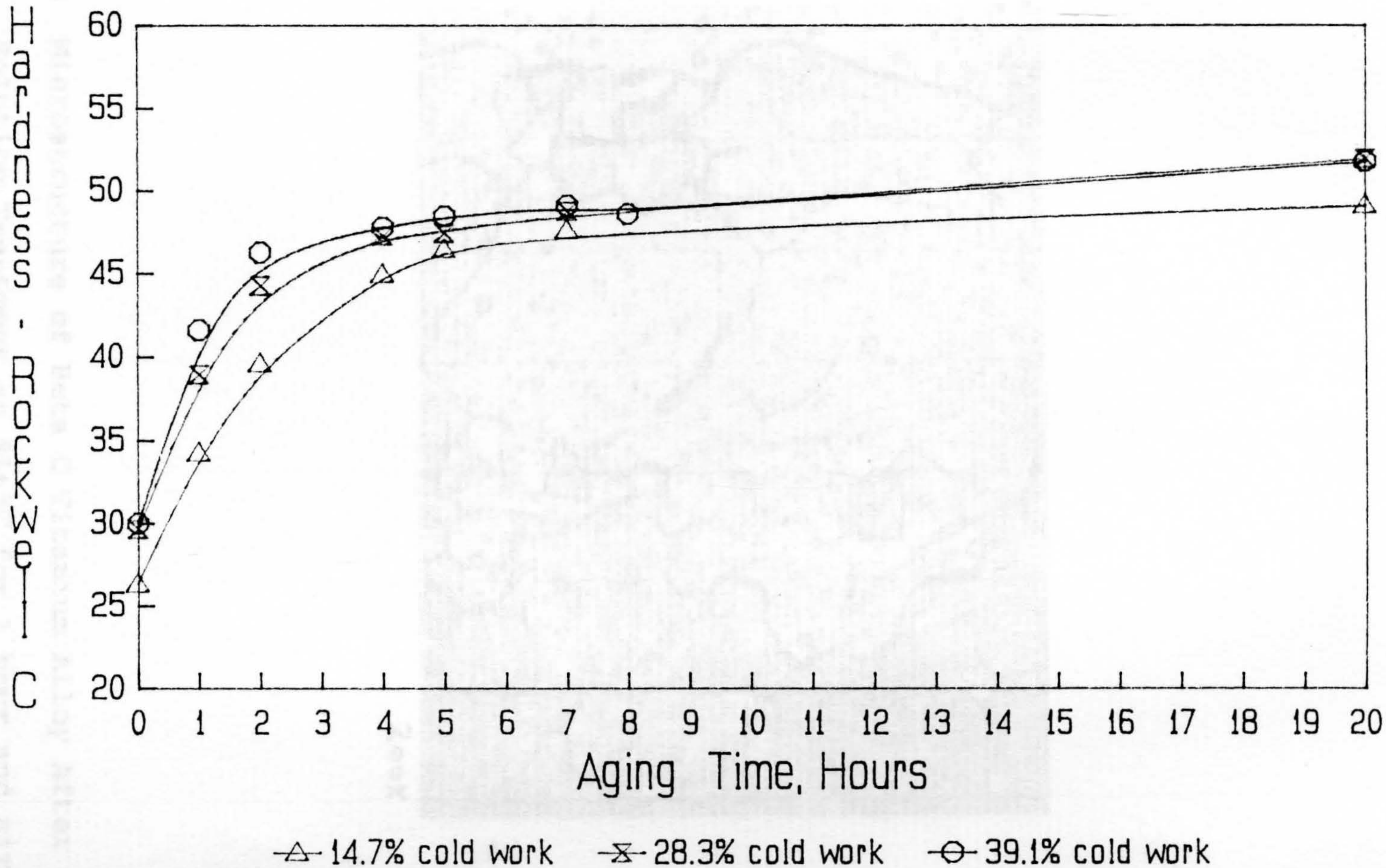
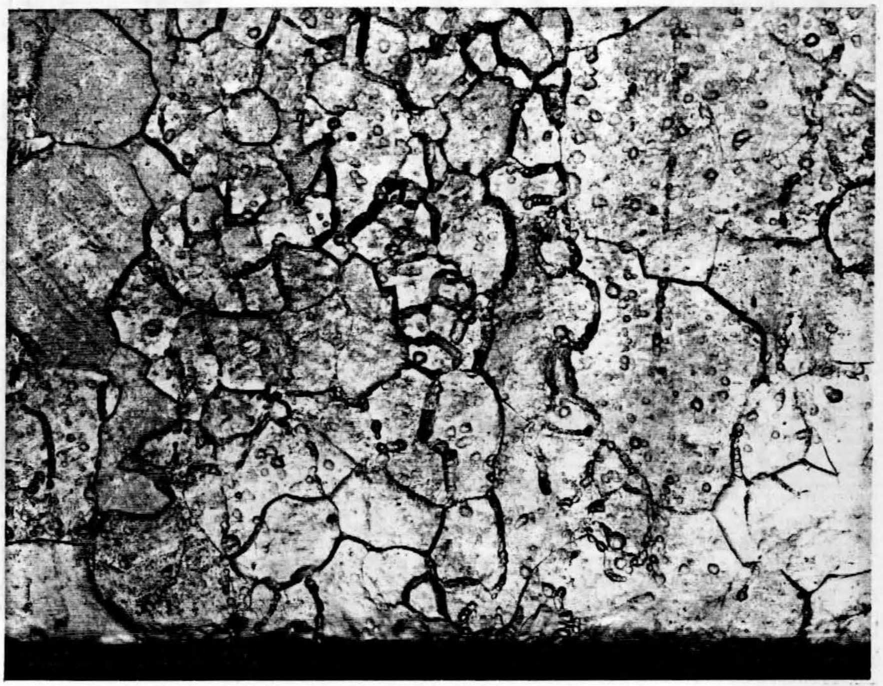


FIG.22 HARDNESS VS AGING TIME AT 427°C



2ooX

FIG. 23 Microstructure of Beta C Titanium Alloy After Solution Treatment at 816°C for 1 hour and air cooled.



2oo X

FIG. 24 Microstructure of Beta C Titanium Alloy After Solution Treatment at 816°C for 1 hour, Air Cooled and Aged at 427°C for 1 hour.

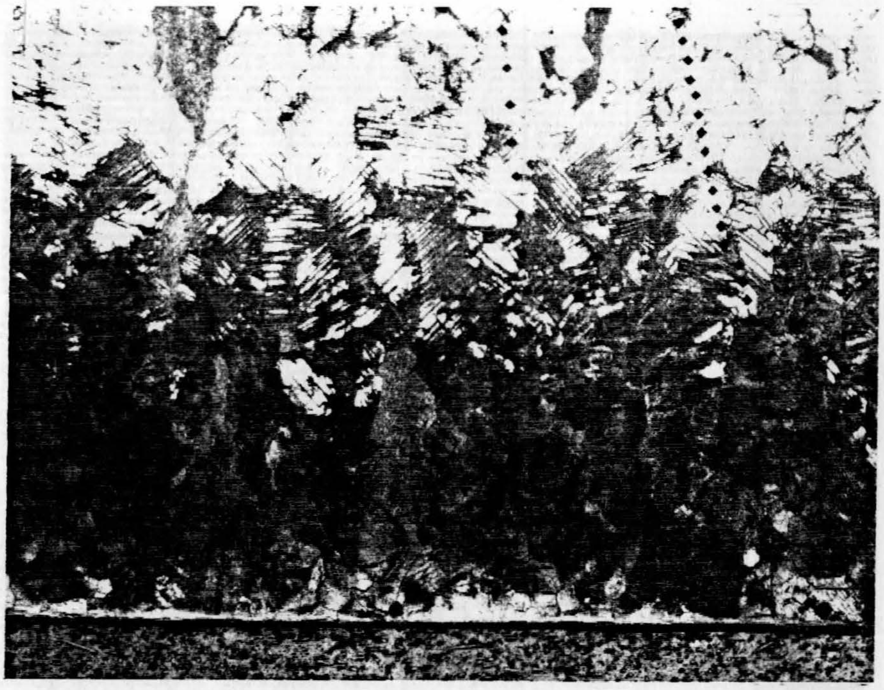
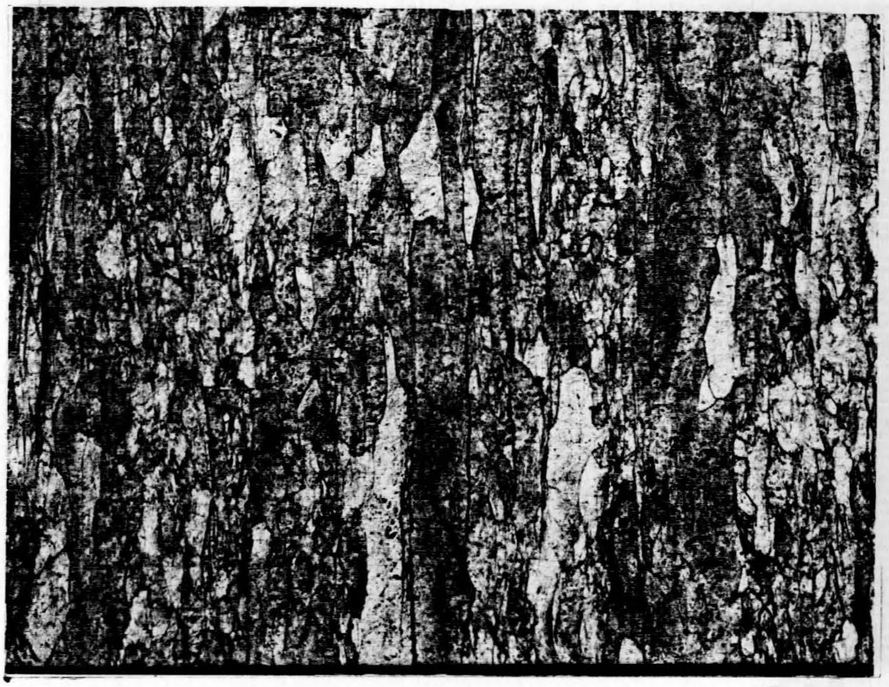


FIG. 25 Microstructure of Beta C Titanium Alloy After solution Treatment at 816°C for 1 hour, Air Cooled and Aged at 427°C for 7 hours.



50x

FIG. 26 Microstructure of Beta C Titanium Alloy after Solution Treatment at 816°C for 1 hour, Air Cooled and Cold Worked 39.1%.

FIG. 27 Microstructure of Beta C Titanium Alloy After Solution Treatment at 816°C for 1 hr, Air Cooled, Cold Worked 39.1% and Aged at 538°C for 20 hrs.

100X

FIG. 28 Microstructure of Beta C Titanium Alloy After Solution Treatment at 816°C for 1 hr, Air Cooled, Cold Worked 39.1% and Aged at 427°C for 20 hrs.

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