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Partial Report

on

Low Carbon Iron Alloys

NAVAL RESEARCH LABORATORY  
ANACOSTIA STATION  
WASHINGTON, D. C.

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### ABSTRACT

A new class of precipitation hardening iron alloys has been developed which do not require quenching from the solution temperature. These alloys have tensile strengths from 100,000 to 200,000 pounds per square inch and their ductility compares favorably with that of heat treated S.A.E. steels of the same strength. These alloys may be used for structures which are too large or complicated for easy heat treatment and for heavy armor.

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## INTRODUCTION

### (a) Authorization

1. This investigation on carbon free iron alloys was carried out under Ordnance Project Order O311-Ord. of 13 November 1939.

### (b) Statement of Problem

2. The purpose of this investigation was to determine whether carbon free alloys may be developed which have desirable properties and characteristics which are not to be found in steels, that is, iron alloys which contain carbon.

### (c) Known Facts Bearing on the Problem

3. There are several ways in which iron may be strengthened without the use of carbon.

A. The substitution for carbon of some other element which acts in a similar manner, for example, phosphorus, boron, or nitrogen.

B. Any alloying element which goes into solid solution in ferrite will harden and strengthen it. The increase in strength which can be produced by this means alone is limited, for example, one per cent of manganese increases the tensile strength of iron only nine thousand pounds.

C. Alloying elements which bring about a marked decrease in the ferritic grain size may cause a marked increase in strength. For example, a carbon free alloy containing eleven per cent manganese has a tensile strength of 150,000 lbs. (NRL Report No. M-1785).

D. The greatest increase in strength in carbonless iron alloys may be brought about by precipitation hardening. This method was the one selected for investigation.

4. Precipitation hardening (age hardening) is the method used for producing high strength aluminum alloys. Copper beryllium alloys also owe their strength to precipitation hardening. The basis for conventional precipitation hardening is illustrated by the constitutional diagram for aluminum-copper (Plate 1). At 548°C. aluminum can dissolve 5.65 per cent of copper but at 300°C., for example, only 0.7 per cent can be held in solution. The excess copper forms  $\text{Cu Al}_2$  in a more or less finely divided state distributed throughout the alloy. If the particles of this inter-metallic compound are of a certain "critical" size and are uniformly distributed the alloy possesses considerable strength and hardness.

5. To harden a precipitation hardening alloy it is first quenched from the temperature at which the precipitating element goes completely into solution. This quenching treatment makes the alloy soft. It is then heated back to some temperature below the solution temperature and held

long enough for sufficient precipitation to occur to give the physical properties desired. The time which an alloy is held at the hardening temperature depends upon its composition and upon the temperature. Cold working before the precipitation heat treatment increases the rate at which hardening takes place.

6. Conventional precipitation hardening alloys are subject to certain limitations. They must be quenched from the solution temperature. This limits the thickness of material which can be precipitation hardened, since the inside of a piece of metal cools more slowly than the outside. If the piece is too thick so much precipitation takes place during cooling that the center is overaged and brittle.

7. Another trouble encountered with some precipitation hardening alloys is that precipitation occurs preferentially at the grain boundaries instead of uniformly throughout the grains. This results in intergranular brittleness.

8. Several alloying elements are known to produce precipitation hardening in iron. Of these copper is used commercially. While precipitation hardening iron-copper alloys do not need to be quenched, the added strength conferred by the copper amounts to only twenty thousand pounds per square inch. Molybdenum and tungsten make iron precipitation hardening but either 8 per cent of molybdenum or 16 per cent of tungsten are required. Iron may be precipitation hardened also by titanium, beryllium, or by the formation of intermetallic compounds of such elements as nickel and aluminum.

#### (d) Theoretical Considerations

9. If precipitation hardening iron alloys are to be useful they must either have properties not obtainable in iron alloys containing carbon or they must be superior to steels in some other respect. To harden a piece of steel it is necessary to cool it rapidly from a fairly high temperature. This rapid cooling will result in a certain amount of distortion unless troublesome precautions are taken. The depth to which steels harden, even alloy steels, is limited and it is impossible to develop the same strength in a heavy section as that which may be secured on heat treating a small section.

10. Precipitation hardening iron alloys would be useful, then, if they could be made hardenable without quenching. If such alloys did not require quenching, they would also be hardenable in heavy sections.

11. We have invented a class of iron-base alloys which are precipitation hardening without quenching. The principles underlying these new alloys are three:

- A. Many of the alloying elements which cause precipitation hardening in iron are more soluble in austenite than they are in ferrite.
- B. Most precipitation hardening elements do not come out of solution rapidly unless the temperature is above 450°C.
- C. Nickel and manganese lower the temperature at which austenite decomposes.

12. To make a precipitation hardening iron alloy which does not require quenching we combine with a sufficient percentage of the precipitating alloying element or elements enough nickel or manganese (or both) to lower the decomposition temperature of austenite to a temperature at which the rate of precipitation is inappreciable.

13. Other important results are achieved by these means. Precipitation on reheating these alloys to the precipitation temperature is quite rapid due to the very small ferritic grain size resulting from the decomposition of austenite at a relatively low temperature. When an alloy is hardened at 500°C., it may acquire eighty per cent of its added hardness in two hours, be completely hardened in eight hours and lose no hardness when held for over forty-eight hours.

14. Our alloys also appear to be free from grain boundary precipitation. At least they have a fine grained fracture and are not brittle as are alloys which contain the same hardening elements but which have to be quenched.

#### (e) Narrative of Original Work Done at This Laboratory

15. A large number of carbon free iron alloys have been prepared and their hardness determined after quenching, air cooling and furnace cooling, and after reheating to various temperatures and holding for various lengths of time. These alloys include those in which the austenite decomposition temperature was lowered by nickel, by manganese, or by nickel and manganese together. The hardening elements have included aluminum (with nickel), molybdenum, copper (with silicon or aluminum), titanium, and boron.

16. Since the purpose of this study was the development of alloys for structural and ballistic applications, only alloys which can be hardened to give tensile strengths between 100,000 and 200,000 lbs. per square inch have been studied. No attempt has been made, for example, to develop alloys hard enough to machine steel.

17. From the various hardening agents investigated nickel-aluminum was selected for intensive study. The tensile properties of a series of alloys in various conditions of heat treatment were determined and some ballistic tests were made on forged quarter inch plates.

they are on quenching. Those alloys which have the same hardness whether cooled rapidly or slowly develop about the same hardness when given the precipitation treatment. Those which are harder on furnace cooling may show little or no increase in hardness on further treatment.

24. The relationship between the transformation temperature and the tendency to harden on cooling from the austenitic condition is shown in Plate 3, where the difference in hardness between furnace cooled and quenched samples is plotted against the temperature at which austenite begins to decompose. If this temperature is below  $525^{\circ}\text{C}$ . ( $980^{\circ}\text{F}$ .), the alloys show the same hardness whether water quenched or furnace cooled.

25. The time-hardness relationship of these precipitation hardening alloys is similar to that of the conventional precipitation hardening alloys. Two typical alloys, HAV, an Fe-Ni-Al type, and HAP, an alloy in which part of the nickel of HAV has been replaced by manganese, show that at the lower temperatures the maximum hardness attained is higher than that reached at the higher temperatures, although the time required is longer, Plate 4. At  $550^{\circ}\text{C}$ ., the alloys overage rapidly and show a decrease in hardness after two hours; however, at temperatures below  $500^{\circ}\text{C}$ ., the alloys maintain their hardness after 48 hours at temperature.

26. The effect of heat treatment on the mechanical properties of furnace cooled alloys is similar to that of the usual precipitation hardening alloys, Table 3. The tensile and yield strengths increase with time at temperature until maximum values are reached corresponding to the flat portion of the time-hardness curves. However, the elongation and reduction in area do not change with tensile strength in exactly the same manner as steels; thus, they may decrease when the alloys are held at temperature for a long time even though the tensile strength does not change. This decrease in ductility depends upon the composition of the alloy and the temperature employed. Alloy HAV, containing 0.50% aluminum, maintains its ductility and strength regardless of exposure to temperatures below  $500^{\circ}\text{C}$ .. At a temperature of  $550^{\circ}\text{C}$ ., as was shown by the hardening curves, the alloy overages rapidly, so that upon holding for 48 hours the tensile and yield strengths decrease below that of the original furnace cooled values though the ductility is still very good. In alloy HAP, in which the aluminum content (1.86%) is much higher, the ductility is good but the alloy is more susceptible to the heat treating procedure. When the alloy is exposed for long periods of time at temperatures of  $500^{\circ}\text{C}$ . or  $450^{\circ}\text{C}$ ., the ductility decreases. This is shown by a comparison of the properties after holding for 4, 8 and 48 hours at  $500^{\circ}\text{C}$ .. At the end of four hours the properties are good and compare very favorably with those of quenched and tempered SAE steels of the same tensile strength. However, when held for eight hours the ductility decreases and at 48 hours, the alloy is extremely brittle. When the alloy is completely overaged by holding at  $550^{\circ}\text{C}$ . for forty-eight hours, the tensile strength is lowered and the ductility fully recovers.

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27. The tensile strength and impact resistance of some of the alloys are given in Table 4 and the limit velocities in Table 5. To show the ductility of these precipitation hardening alloys in comparison to quenched and tempered SAE steels, the per cent elongation (Plate 5) and the reduction in area, (Plate 6) are plotted against the tensile strength. The alloys possess good ductility in respect to both elongation and reduction in area since they fall above the curve for SAE steels.

28. The ductility of these alloys as measured by the impact resistance is good. The relation between impact resistance and tensile strength (Plate 7) is shown in comparison to similar tests on the nickel steels used in a recent weldability study (NRL Report No. M-1770). The nickel steels are known for their high impact values and, as seen in Plate 7, the precipitation hardening alloys exceed even these values.

29. The photomicrograph of alloy HAV after furnace cooling from the solution temperature and holding for 4 hours at 500°C. fails to reveal any grain boundary precipitation even at 1500 magnification (Plate 8). Alloy HAX, however, which contains an excess of aluminum in respect to the nickel content, shows a precipitate in the grain boundaries after holding for 6 hours at 500°C. This alloy was brittle in this condition but when it was completely overaged by exposing for 16 hours at 550°C., the precipitate coalesced into large particles (as is shown by the photomicrograph,) and the ductility tended to recover.

#### CONCLUSIONS AND RECOMMENDATIONS

##### (a) Facts Established

30. Precipitation hardening alloys have been devised that can be hardened without water quenching. In fact, these alloys may be cooled very slowly without impairment to the mechanical properties.

31. These alloys have excellent ductility and impact resistance in comparison with heat treated SAE steels of the same strength.

32. The limit velocities given in Table 5, while only 2-1/2% below the average value of limit velocities of good STS of the same hardness, probably do not represent the best values for these precipitation hardening alloys, since the test plates were made from small experimental heats and forged with perhaps not too much control. Furthermore, the effect of heat treatment on ballistic properties was determined for a limited number of plates only and the best combination of solution temperature and hardening time and temperature was probably not found.

##### (b) Opinions

33. Since these precipitation hardening alloys may be hardened without quenching, heavy sections and structures difficult to heat treat because of shape may be heat treated.

34. Although the alloy content of these precipitation hardening alloys is higher than that of STS, the cost for alloying elements is more than offset by the lower cost of heat treatment. Furthermore, since part of the nickel content may be replaced by manganese, no new burden need be placed upon the nickel supply.

35. The fact that these alloys may be machined while soft and then hardened with little or no distortion should greatly aid in the manufacturing process.

#### RECOMMENDATIONS:

36. The precipitation hardening alloys appear to have two definite types of applications.

- A. Structural members which are difficult to heat treat because of size or complicated shape. After machining, complicated structures may be hardened with little or no distortion, since the precipitation temperature 450-500°C. is comparatively low.
- B. Heavy Armor. Since these alloys do not have to be quenched, uniform properties may be developed throughout thickness as great as twenty-four inches.

37. Before definite recommendations can be made more experimental work should be done.

- A. Preliminary work on alloys of Fe-Ni-Ti, Fe-Ni-Cu-Si, Fe-Ni-Cu-Al, Fe-Ni-Mo, and Fe-Mn-Ti, has shown that these may be hardened in the same manner as the Fe-Ni-Al alloys. It is not known whether the properties of these alloys are better than those of the Fe-Ni-Al alloys, but some of these alloying elements may be more available than nickel and aluminum.
- B. These alloys would be easier to make commercially if the carbon content can be as high as 0.10% without impairing the properties.
- C. These alloys were not intended for use as light armor since carbon steels with little or no alloying additions may be used satisfactorily. Since, however, these alloys are intended for heavy armor, more work should be done on quarter inch and thicker sections to obtain the best heat treatment and the behavior in heavy sections should be studied.
- D. A study should be made of the weldability of the alloys.

TABLE 1  
Chemical Composition

Alloy	Ni	Mn	Al	Si	C
HE	7.88	0.18	1.38		0.02
HH	7.65	1.48	1.65		
HK	7.50	0.19	2.75		
HN	5.65	3.04	1.10		
HAE	7.85	0.23	0.62	0.11	0.03
HAF	7.38	0.30	1.13	0.19	0.01
HAH	7.34	0.31	2.98	0.24	0.01
HAI	3.84	0.31	1.96	0.22	0.01
HAK	1.75	3.55	1.89	0.25	0.01
HAL	1.64	7.53	1.84	0.27	0.02
HAM	3.67	5.28	1.86	0.26	0.01
HAN	7.81	0.30	0.68	0.04	0.03
HAO	7.94	0.38	1.57	0.13	--
HAP	3.18	2.76	1.84	0.18	--
HAR	9.66	0.40	0.69	0.12	--
HAS	10.20	0.44	1.47	0.19	--
HAT	5.13	2.08	1.64	0.10	0.04
HAV	11.97	0.41	0.50	0.18	--
HAW	11.99	0.40	1.50	0.30	--
HAX	10.09	1.80	1.66	0.11	--
HAY	2.14	4.52	1.58	0.17	0.04

TABLE 2

Alloy	Hardness - Rockwell "C"			Maximum Hardness Obtained on Holding at 500°C. (932°F.)			Composition			Transformation Temperature °C.
	As			Water Quenched	Air Cooled	Furnace Cooled	Ni	Mn	Al	
	Water Quenched	Air Cooled	Furnace Cooled							
HE	22	21	22	40	37	--	7.88	0.18	1.38	592
HH	24	24	36	46	40	--	7.65	1.48	1.65	
HK	21	37	31	45	42	35	7.50	0.19	2.75	
HN	25	29	28	44	42	39	5.65	3.04	1.10	
HAE	19	18	17	25	24	24	7.85	0.23	0.62	594
HAF	21	22	31	36	34	34	7.38	0.30	1.13	608
HAH	24	39	33	47	43	37	7.34	0.31	2.98	721
HAJ	20	17	24	36	32	35	3.84	0.31	1.96	774
HAK	24	24	29	43	41	41	1.75	3.55	1.89	400
HAL	26	23	28	44	44	43	1.64	7.53	1.84	274
HAM	27	25	27	44	44	43	3.67	5.28	1.86	377
HAN	18	16	15	32	29	28	7.81	0.50	0.63	586
HAO	26	32	36	42	40	38	7.94	0.38	1.57	634
HAP	24	22	26	39	37	36	3.18	2.76	1.86	562
HAR	22	22	23	34	32	31	9.66	0.40	0.69	466
HAS	24	26	38	44	42	41	10.20	0.44	1.44	482
HAT	23	25	33	43	41	39	5.13	2.08	1.64	580
HAV	22	20	21	35	33	32	11.97	0.41	0.50	336
HAW	25	24	27	44	41	42	11.99	0.40	1.48	475
HAX	25	24	30	48	47	45	10.09	1.80	1.66	438
HAY	23	22	23	35	34	33	2.14	4.52	1.58	452



TABLE 4

Mechanical Properties

<u>Alloy</u>	<u>Tensile Strength</u>	<u>Yield Strength 0.2%</u>	<u>Percent Elongation</u>	<u>Reduction in Area</u>	<u>Charpy Impact</u>	<u>Treatment</u>
HE	135000	118000	22.5	59.5		A.C.* 450°C. - 2 hrs.
HJ	128000	109000	25.0	64.0		W.Q.** 500°C. - 2 hrs.
HU	95400	82000	22.5	64.0		A.C. 450°C. - 2 hrs.
HN	157000		13.8	63.0		A.C. 540°C. - 2 hrs.
HX	145000	128000	20.0	55.0		F.C.*** 500°C. - 2 hrs.
HAN	126000	108500	23.75	59.0	92	F.C. 500°C. - 8 hrs.
HAO	150000	132000	21.25	57.0	15	F.C. 450°C. - 8 hrs.
HAP	152000	133000	20.0	59.0	10	F.C. 500°C. - 4 hrs.
HAR	152000	142000	21.25	61.0	25	F.C. 500°C. - 48 hrs.
HAT	152000	136000	16.25	49.0	5	F.C. 500°C. - 4 hrs.
HAV	150000	136000	21.25	62.0	30	F.C. 500°C. - 4 hrs.
HAW	197000	165000	17.9	46.0	3	F.C. 500°C. - 4 hrs.
HAY	140000	125000	20.0	56.0	19	F.C. 450°C. - 8 hrs.

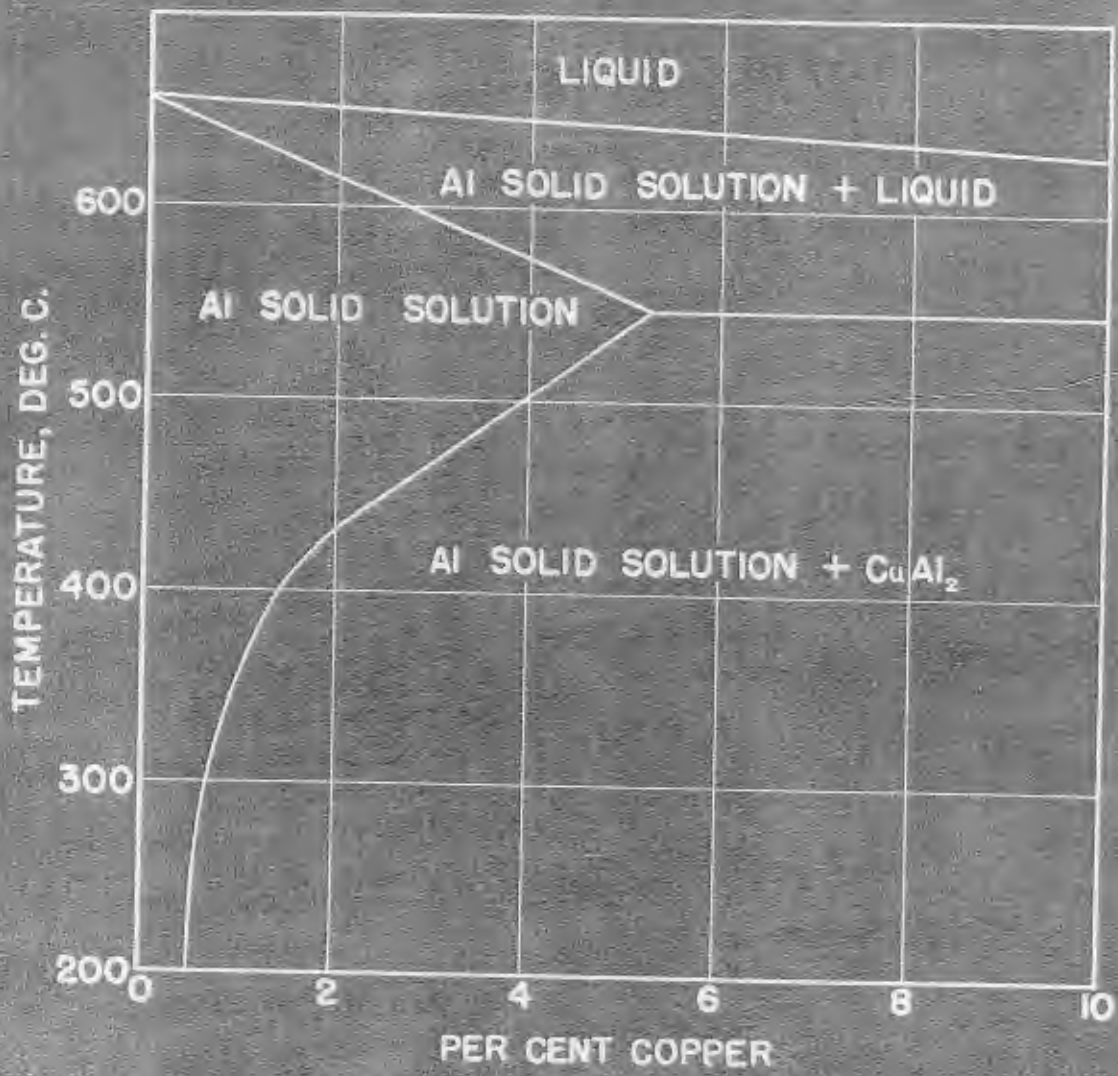
\* Air cooled  
 \*\* Water Quenched  
 \*\*\* Furnace Cooled.

TABLE 5

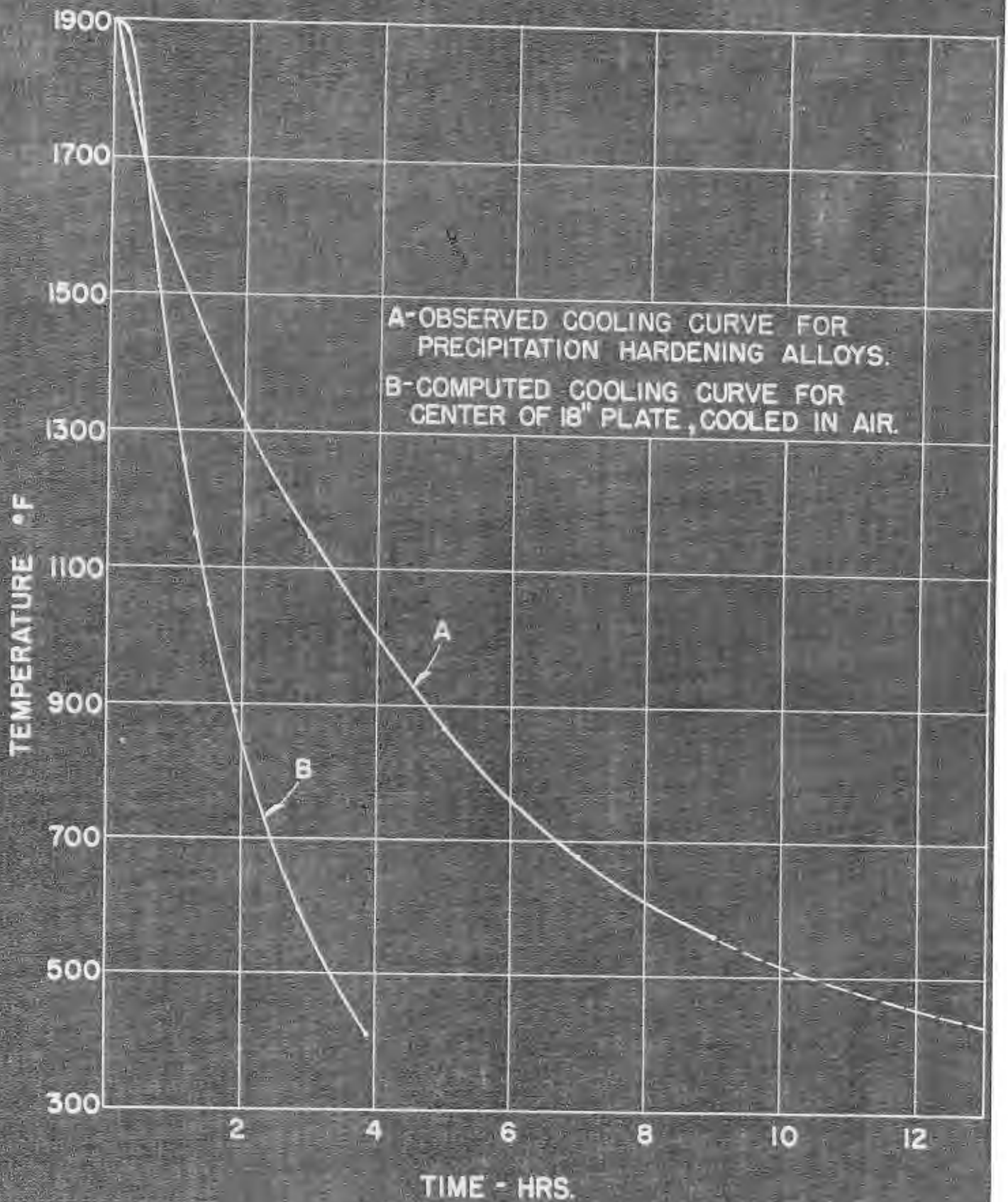
Ballistic Properties

<u>Alloy</u>	<u>Heat Treatment</u>	<u>Hardness Brinell</u>	<u>Plate Thickness</u>	<u>Limit* Velocity-ft/sec.</u>
HAO	Furnace cooled + 4 hrs. at 600°C.	302	0.25	1540
HAR	Furnace cooled	269	0.251	1460
HAR	Furnace cooled + 2-1/2 hrs. at 500°C.	293	0.244	1520 ± 25
HAS	Furnace cooled + 9 hrs. at 600°C.	302	0.249	1555
HAV	Furnace cooled + 2 hrs. at 450°C.	285	0.251	1479
HAV	Furnace cooled + 4 hrs. at 500°C.	302	0.243	1520
HAW	Furnace cooled + 12 hrs. at 600°C.	302	0.247	1559
HAW	Air cooled + 1-1/2 hrs. at 500°C.	293	0.250	1550 ± 25
HAX	Furnace cooled + 3/4 hrs. at 600°C.	285	0.250	1547
HAY	Furnace cooled	263	0.252	1490

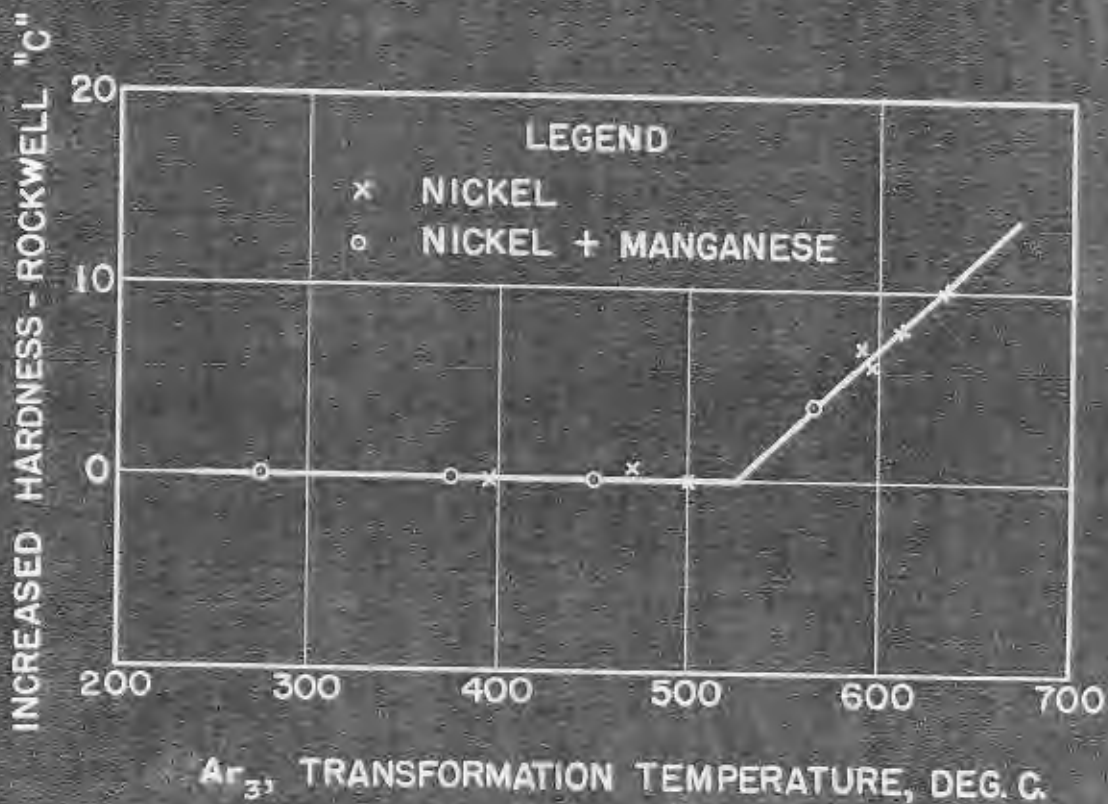
\*Except where otherwise indicated,  
estimated gross error of limit  
velocity is ± 20 ft./sec.



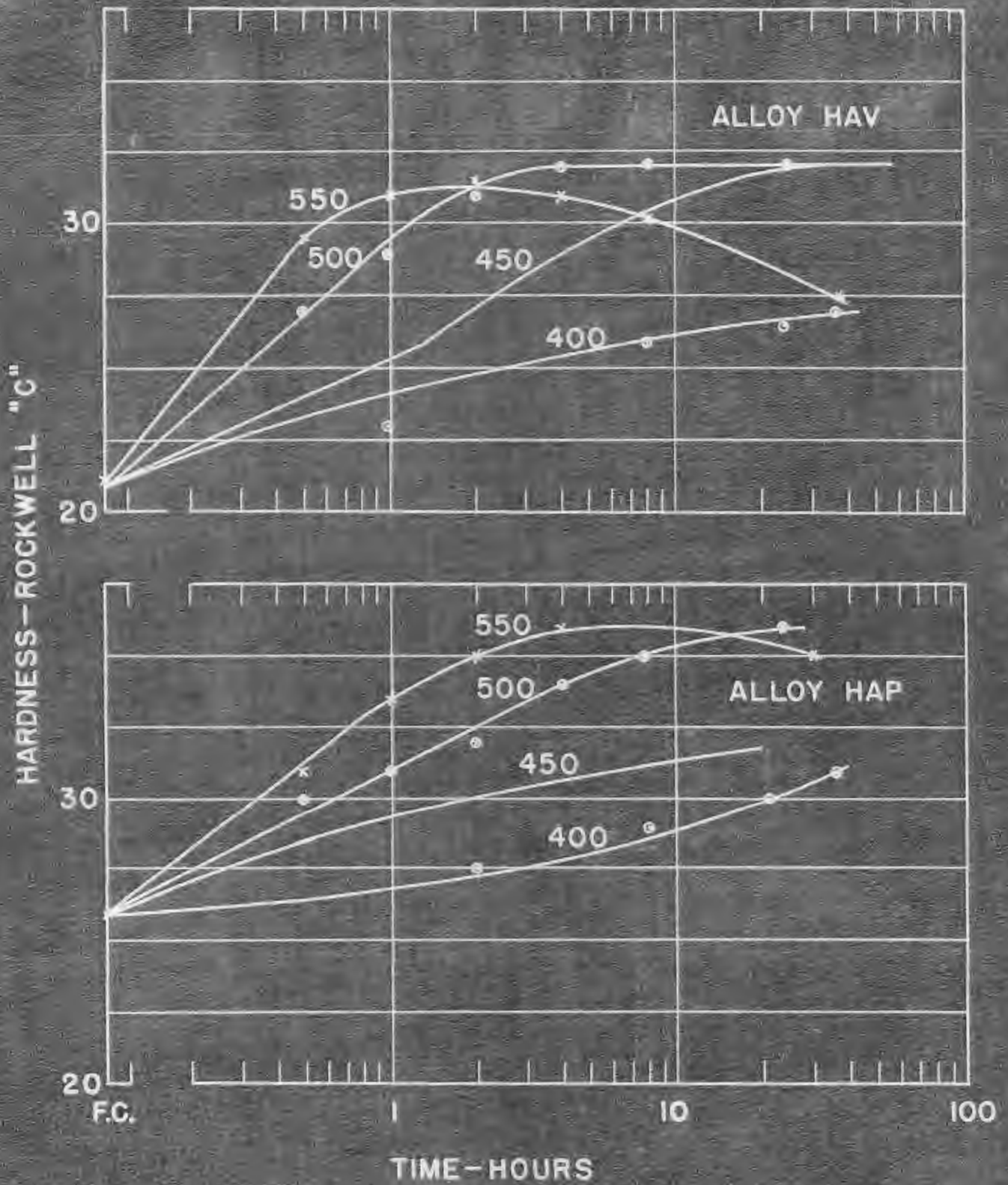
ALUMINUM - COPPER CONSTITUTIONAL DIAGRAM

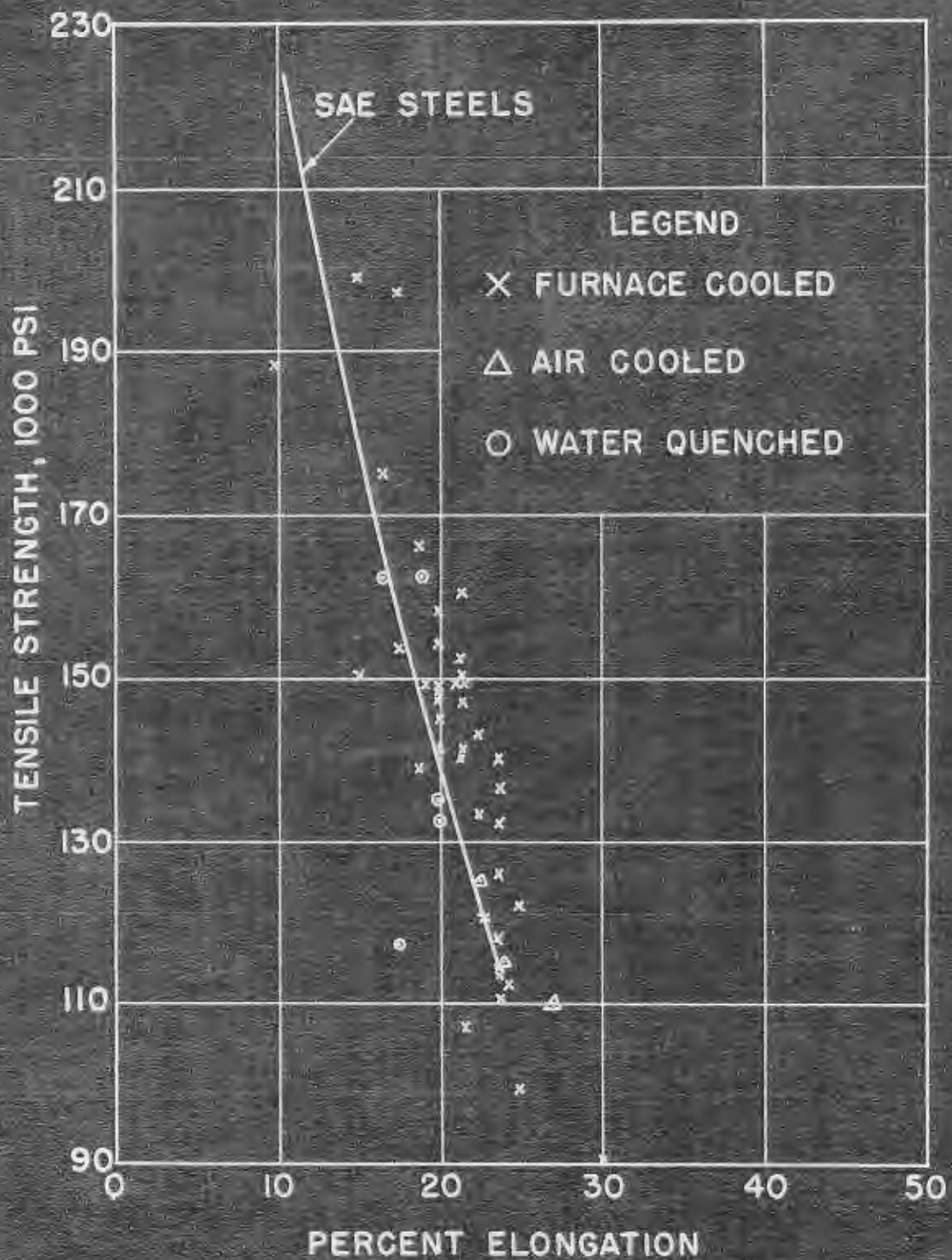


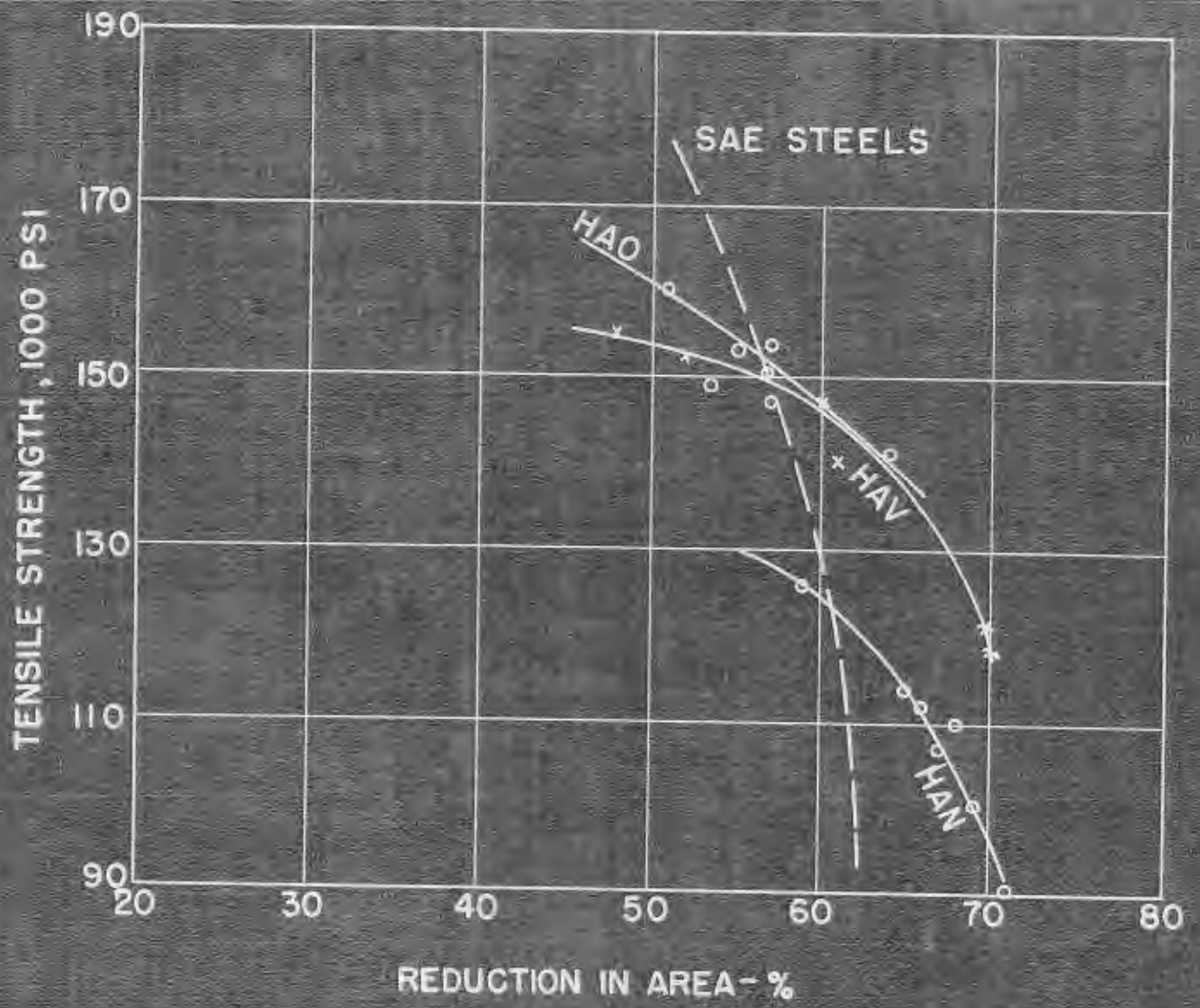
INFLUENCE OF TRANSFORMATION TEMPERATURE  
ON INCREASE HARDNESS BETWEEN WATER  
QUENCHING AND FURNACE COOLING.



# HARDENING CHARACTERISTICS AFTER FURNACE COOLING

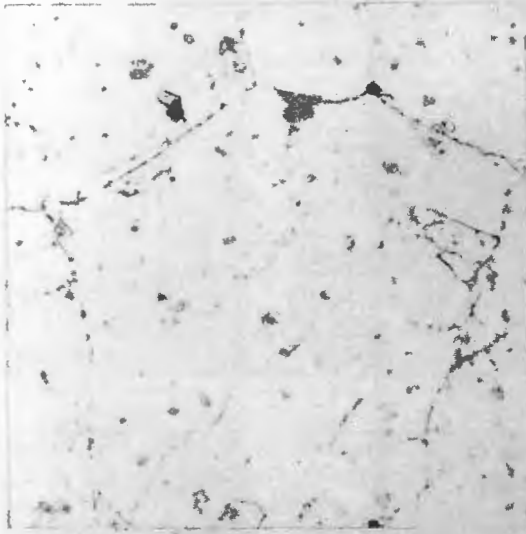








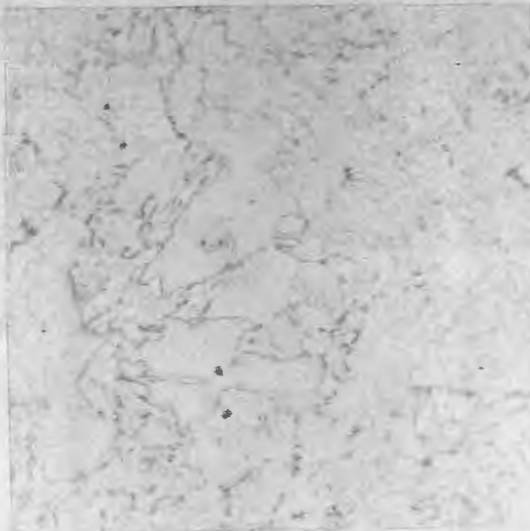
# MICRO-STRUCTURE AFTER PRECIPITATION HARDENING TREATMENT



ALLOY HAX  
WATER QUENCHED AND HELD  
FOR 6 HOURS AT 500°C.  
1500X



ALLOY HAX  
WATER QUENCHED AND HELD  
FOR 16 HOURS AT 500°C.  
500X



ALLOY HAV  
FURNACE COOLED AND HELD  
FOR 4 HOURS AT 500°C.  
1500X