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THE EFFECT OF HEAT TREATMENT TEMPERATURES (ANNEALING, AGING AND DOUBLE AGING) ON HARDNESS OF 16%NI CAST MARAGING STEELS.

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Abstract

The primary purpose of the present work was to study the effect of different annealing and aging temperature on the mechanical properties of 16%Ni cast Maraging steels. In addition subsidiary studies were made of the hardening mechanism responsible for high strength. It was found the annealing temperature must be done above approximate 750C° but not exceed 1250C° to give the good hardness during aging.. Marked hardening occurred within several minutes during from 485°C to545 °C but only after several hours in the range between 325 to 375 °C Heat treatment of 3 hour at 485 °C of cast alloys was most satisfactory. While the aging at 485 °C for long time decrease this hardening caused softening due to the reversion of the body centered cubic matrix to austenite. The aging kinetics mechanism refer to that lattice defect were influencing the reaction rate. X-ray diffraction evident indicated the formation of any bcc structure of matrix as martensite and the appeared that hardness was due to coherent precipitate having concentration in matrix that formed as Fe2CoNi,Ni3Ti and Ni3Mo phases. The double aging of cast alloys in different temperature given normal hardening result.

ملخص البحث

ان الغرض الاساسي من هذا البحث هو دراسة تاثير تغير عدة درجات حرارية للمعاملة الحرارية (التلدين والتعتيق) على صلادة مسبوكة الفولاذ المراجيني(المارتنسايت المعتق) الذي يحتوي على (16%Ni). اضافة الى دراسات ثانوية تخص ميكانيكية استجابة السبائك المصلدة الى المتانة العالية. لقد وجد ان درجات حرارة التلدين يجب ان تنجز فوق درجة حرارة (750°C) وان لاتتجاوز (1250°C)لاعطاء صلادة جيدة ومقبولة خلال التعتيق كذلك وجد بداية ظهور الصلادة بعد عدة دقائق خلال التعتيق في (485°C) الى(545°C) بينما هذاالظهور يبدا بعد عدة ساعات في مدى درجات حرارة(325) الى (375°C) . المعاملة الحرارية التعتيق لمدة (3 hr) عند (485°C) للسبائك المسبوكة يكون مقنع في اعطاء قيم صلادة جيدة ولكن التعتيق لفترة طويلة عند هذه الدرجة الحرارية ربما يسبب في انخفاض في قيمة الصلادة ناتجه من تلين التركيب بسبب ظهور انعكاس تحول المارتنسايت (bcc) الى اوستنايت . انة ميكانيكية الحركية للتعتيق تشير الى ان العيوب الشبكية متاثرة بمعدل التفاعل خلال التعتيق حيث ان فحص حيود اشعة (X) يوضح مؤشرا تشكيل تركيب قوام ذو (bcc) بشكل مارتنسايت اضافة الى ان ظهور زيادة في الصلادة ناتج من تمركز رواسب التصاقية في القوام والتي تشكل بصيغة اطوار مثل (Fe3CoNi,Ni3Ti and Ni3Mo). التعتيق المزدوج للسبائك المزدوج للسبائك المسبوكة عند عدة درجات حرارية مختلفة تعطي نتائج تصليد اعتيادية .

Introduction

The discover of wrought 18%Ni Maraging steel has provided class of alloys possessing high strengths and toughness [1].These steels contain 18%Ni and 7-9%Co,3-5%Mo,0.2-0.8%Ti and less than 0.03%C max . They are austenitic at elevated temperature and transform to martensit on cooling to room temperature. The transformation characteristics illustrated in Fig.(1) are form by diagramly Jones and Pumphery[2].Within the limits of martensite in body centered cubic. Reheating the steel to 480 ^{o}C the Maraging treatment produced 1400-1900 Mps yield strength, notch tensile strength/tensile strength ratio over 1.4 and up to 60% reduction in area.

It was believed that the industrial needs for high strength cast alloy with high toughness could be filled by cast Maraging steels .Therefore the more studies included cast Maraging steel.The 17%Ni 1600 grad developed for cast has slighting different composition compared with wrought grades in table(1)[3,4,5]. The 17%Ni 1600 cast Maraging steel grade of nickel Maraging is normally annealed by homogenizing for 4hour at 1150 followed by air cooling. Maraging of the solution annealing material is effected by heating 3 hours at 480C$^{\circ}$ [6, 7].Typical mechanical properties after these treatments are presented in table (2) [6]. The more studies included Maraging steel. Some studied on heat treatment of wrought Maraging steel included ,Floreen and R.F.Decker study the effect of heat treatment and thermo-mechanical treatment of 18%Ni wrought Maraging steel in 1979[8]. Stiller and Daniox study concerned evolution of Mo –rich precipitates of wrought Maraging steel (IRK91) with composition 12%Cr,4%Mo,9%Ni and 2%Cu was investigated after heat treatments at(475 ^{o}C)up to400 hr using atom probe field. The investigation reveled that nucleation of a Mo-rich phase stars between 1 and 2hr of aging at matrix near the Ni- rich precipitates that were found earlier and at the martensite boundaries[9]. The aging behavior of 18Ni(350) maraging steel was studied by Mossbauer spectroscopy. The aging reactions start with the redistribution of atoms, which results in the formation of Fe-Co-rich zones and Ni-Mo-Ti-rich zones at the early stages of aging. The redistribution of atoms is fast at the initial stages of aging and then is slow. The shape of the concentration fluctuation during the early stages of aging is close to a pulse wave. Intermetallic compounds precipitate on the Ni-Mo-Ti-rich zones.[10] . The evolution of precipitates in maraging steel of grade 350 was studied using the complementary techniques of small angle X-ray scattering (SAXS) and transmission electron microscopy (TEM). These investigations revealed that ageing the steel at 450 ^{o}C involved a rhombohedral distortion of the supersaturated b.c.c. martensite accompanied by the appearance of diffuse omega-like structures.

This was followed by the appearance of well-defined omega particles containing chemical order. At the ageing temperature of 510 ^{o}C, Ni-3(Ti, Mo) precipitates were the first to appear with a growth exponent of 1/3. [11].

The age hardening kinetic in temperature range of 713 to 813 K a 2400 Mpa grade coblet- free maraging steel (Fe.18.8-19.1%Ni ,4.4-5.4%Mo,2.6%Ti)has been studied by YIHE, KE YANG and others. The study included microstricture and mechanical properties that showed a high number of(Ni3Ti) and (Fe2(Mo,Ti)) precipitates were formed during the aging process[12]. The effect of the heat treatment included solution treatment and aging treatment on the proire austenite grain size microstrcture and mechanical properties of a precipitates hardening maraging stainless steel was investigation by KAILIV,YIYIN SHAN and others [13].

Experimental procedure

The composition of the cast alloys studied was the follows 69.77%Fe, 0.02%C,16.34%Ni,4.85%Mo,8.65%Co,0.6%Ti,0.13%Al,0.12%Cr,0.39%V and traces contains of Zr,Cu,Si and Mn.

The alloy was mad as a (4Kgs) charge that melted using a high frequency rapid induction melting furnace supple as high as (60 Kw) at nominal frequency of (19.6 KHz)from (415-440 V). A standard melt super pouring temperature of (1600±10 ^{o}C) was maintained to achieved a pouring temperature (1500±10).The material used in the present study was taken from product of sand casting as solid bar shape with dimensions(Ɵ25*200mmlength) that shown in Fig(2). The solid bar has homogenization treatment at 1150^{o}C for one hour and air cooling to room temperature. The alloys were studied by Vickers hardness measurement augmented by tensile test and structured studies. Preliminary work had shown that the hardness values correlated quit well with yield strength. Thus the hardness measurement furnished a simple and reasonably reliable method of evaluating the mechanical properties.

The effect of annealing temperature on tensile strength were studied on samples which cutting as bar according DIN 50125-As∗25 and annealed for 1 hr at temperature range from 650 to 1250 ^{o}C. The aging behavior of the alloy after these various initial treatments were then evaluated by heat treated the samples in the temperature range of 325 to 545 ^{o}C for times ranging from half min to several hours.

Most of the heat treatments were done in nitrogen atmosphere in vacuum furnace except for that short time aging tests which were done in salt bath to insure rapid heating. All of the specimens were gas cooled as prior tests had indicated no effects due to cooling rate. A number of light examination and X-ray diffraction analyses also used.

Results and Discussion

Annealing Behavior

In this project the annealing treatment occur on cast alloy structure at different temperature.

When the cast alloy was annealed at 650 ^{o}C the structure contained approximately35% austenite after cooling to room temperature. This austenite had the fine, lamellar appearing structure as shown in Fig. (3). Apparently at this annealing temperature the austenite that found during annealing became partially stabilized and did not retransform to body-centered cubic structure on cooling to room temperature. This retained austenite may have been due to transformation of the metastable body centered cubic martensite(α2) matrix found on cooling into the equilibrium ferrite (α)and austenite(γ) phase asX-ray diffraction shown. During this transformation there was probably some partitioning of the alloying elements between the α and γ phases so that some of the austenite was enriched and did not transform back to α2 on cooling to room temperature.

When the annealing temperature was raised the amount of austenite retained on cooled to room temperature decreased until only the body-centered cubic phase was present. The minimum annealing temperature required to eliminate all the austenite was approximate 750 ^{o}C.At temperature 650 ^{o}C and below. When reheated a cast sample for this temperature for a period long enough to establish equilibrium ferrite of composition α and austenite of the composition (γ) are formed from the previously existing martensite. It is seen that the ferrite is leaner in nickel and the austenite is richer in nickel that the prior martensite. On cooling back to room temperature the ferrite does not transform to martensite. In addition the austenite may be sufficiently enriched with nickel that it becomes stable and therefore does not transform to martensite. To return the austenite to transformation condition. It necessary to reheat to high temperature more than 650 ^oC that lie the materials in the single phase gamma region where the austenite can be restored to it original composition.

When aging this above specimens the result appears eliminate this austenite to achieve satisfactory hardening during aging. The martensite is perfect at aging after annealing temperature 825 ^oC the microstructure in Fig. (4) Appear this structure. The austenite grain size was No 6 to 8 according the ASTM after annealing at temperature to 980 ^oC. Above this temperature grain growth accorded with ASTM grain size No 0 after at 1250 ^oC.

With high annealing temperatures a widmansttaten morphology was evident in the microstructures of the body centered cubic α2 phase as in Fig.(5) while as explain in above the annealing at 825 ^oC appear the microstructure have100% bcc with low amount of widmanstatten structure. As the structure in Fig.(4),(5) were entirely body centered cubic the X-ray diffraction shown no differences in chemical composition between the dark plates and light back ground in area. Also the micro hardness appears no different between two areas in Fig.(5). The tensile properties after annealing at 825 ^oC to 1250 ^oC are listed in table 3 also the tensile properties obtained after various heat treatments are included in table(3). The general findings were in accord with the hardness-1hr at 825 ^oC gave yield strength of 1434Mpa.Raising the annealing temperature to 875 ^oC lowered the annealing 1350 ^oC. Further increase of the annealing temperature had little effect on strength.

Aging behavior

The hardness of the specimens initially annealed at 825 ^oC and then aging are shown in Fig .(6). More extensive data for specimens initially annealed at 985 ^oC are shown in Fig. (7). The curves show a rapid initial rate of hardening at 485 to 545^oC while the response at 325 to 375^oC was slower. The general shapes of these curves are comparable to these of precipitation hardenable alloys. In the present alloy, however over aging is the conventional sense may not occur because have short time for aging reached to 48 hrs. The think that the hardness will drop with prolonged aging because that reformation of austenite .The general appearance of the austenite form after prolonged aging was similar to the fine lamellar type of the in Fig.(3). The amount of austenite that was observed after any given aging treatment varied somewhat with the initial conditions. Prior amount of austenite in higher annealing temperatures reduced the austenite content.

The effects of various initial annealing treatments on the hardness after aging at 485^oC are shown in Fig. (8). Changing the initial condition of the alloy prior to aging produced some distinct changes in hardness behavior. The specimens that annealed at 650^oC contained about 35% austenite and were not hardening by the aging treatment. Raising the annealing temperature from 825 to 1250 ^oC resulted in lower hardness after aging.

There is still broad field in annealing and aging conditions to yield reproducible high strengths. First the stabilization of austenite by annealing below 735 ^{o}C or by aging to high temperature should be avoided to obtain optimum strength.

There also the aging treatment have considerable field because of the relatively flat nature of the hard time curve at 485 ^{o}C. For most purposes aging for 3hr at 485 ^{o}C would be the normal heat treatment but varying the aging time at 485 ^{o}C from 2 to 6hr does not significantly alter the hardness. Aging could also be done at other temperature. But the increase in the rate of revelation to austenite at higher temperatures and sluggishness of reaction at lower temperatures. Indicated that 485 ^{o}C ±10 is probably the optimum aging temperature for cast alloy. Strength can be lowered by higher annealing temperature and or aging at lower temperatures. For application requiring elevated temperature strength the use of higher annealing temperatures is advantageous in retarding austenite reversion and thus maintaining the strength properties for longer time periods. Some gain in strength was also noted by aging at435 ^{o}C for 24hr.

The X-ray diffraction evident indicated as in Fig (10) the strength occurring above alloys on aging at 480 ^{o}C results from the early formation of zones or clusters based on an Ni3Mo grouping containing iron as (Ni,Fe)3Mo which at higher aging temperatures may give way or evolve into a precipitate of Fe2Mo . At the lower aging temperatures and long holding times the clusters may perhaps be supplemented by the Fe2Mo precipitate. The third precipitate containing titanium forms in the promotion of age hardening in these cast steels this precipitate is FeTi sigma phase.

Double Aging

Several experiments were conducted to study the effects of double aging heat treatment specimens were initially aging at one temperature and then aging for various times at second temperature. The result showed that when the specimens were aging at lower temperature and then at 485 ^{o}C the resultant hardness curves at 485 ^{o}C closely followed the normal 485 ^{o}C aging curve .Of more interest are the results in Fig.(9) showing that when specimens were first aging at 435 or485 ^{o}C to produced about 530 Vickers hardening and then held at 375C ^{o}C there was essentially little change in hardness after 48hr at 375 ^{o}C. The curves in fact crossed that obtained as a result of aging only at 435 ^{o}C from above results excluded the results indicated that there is no special advantage to be gained by double aging treatments. One possible benefit of the apparent stability of the aging structure at lower aging temperatures might occur in elevated temperature service applications in the 375 ^{o}C. In such cases the alloy when aging at 485 ^{o}C might not be susceptible to the long-time hardening and embitterment that occur in many alloys.

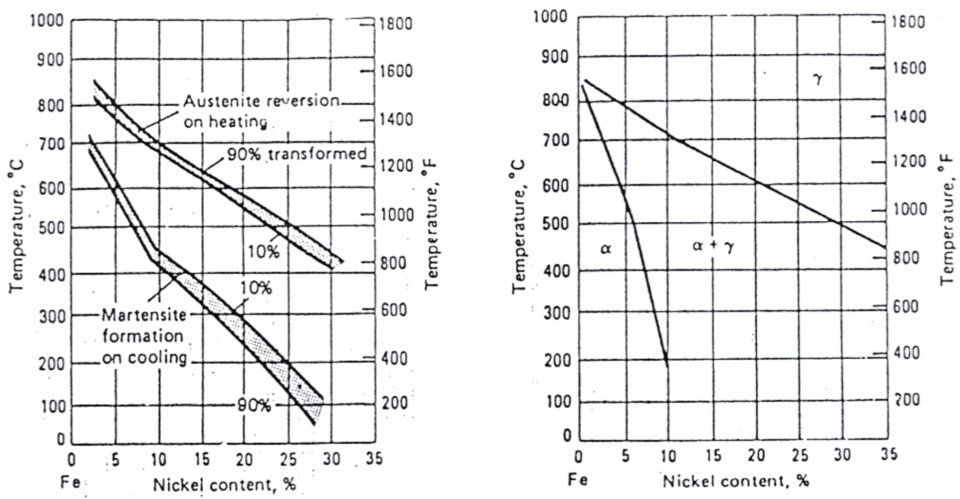
Over Heat after Aging

When the cast alloys samples are heated for extended period of time at higher aging temperature the over aged of matrix tends to revert to austenite. The same thing happens on heating at temperatures between the aging range and the annealing range. The results of these tests listed in table(4) indicated that the original hardness was essentially restored by re-annealing and re-aging. With over heating there was a progressive decrease in hardness with increasing time and temperature. The extent of the reversion depends on the time and temperature, thus the aging 10hr at 485 ^{o}C which they considered to started of reverted austenite. Greater amounts of the phase were seen in material aged 48hr at the same temperature. The X-ray diffraction study indicated the presence of approximate 12 to 14% austenite in these specimens. 3% austenite was found in cast steel aged 6 minutes at 545 ^{o}C, where as 6% austenite was developed by extending the age time to 30 minute. We thinks the amount of austenite increased and then deceased with increasing temperatures probably because of changes in stabilization of the austenite thus at the lower overheating temperature

The austenite that formed was probably due to the α2 to α+ (γ) reaction and was sufficiently enriched in alloy content so that it did not transform back on cooling. At higher over heating temperatures the austenite probably formed by the α2 to (γ) reaction and most of the austenite transformed back to α2 on cooling to room temperature. However when the specimen were re-annealed and re-aged the hardness returned substantiating to the average initial hardness before over heating.

Conclusions

1- The annealing temperature of 16%Ni cast Maraging steels must be occur in range 825±10 ^{o}C and the rang of 600 to 700 ^{o}C must be avoided to prevent retained austenite.
2- Marked hardening during aging occurs in several minutes at 485C or 545 ^{o}C. and optimum treatment occur after 3-6 hr at 485 ± 10 ^{o}C.
3- Larger amounts of retained austenite are present after over aging that appear after 5 to 30 minute in range 545 to750 ^{o}C.The initial hardening can be restored by re-annealing and re-aging.
4- The results of double aging appear that the aging at lower temperature followed by a second aging at 485 ^{o}C produces hardness value equal to values produced by a single aging at 485 ^{o}C and the specimens first aged at 485 ^{o}C or 435 ^{o}C and then aged at 375 ^{o}C no change in hardness after 48 hr.
5- Electron diffraction studies of the aging cast gave evidence of phase (Fe2CoNi and Ni3Ti, Ni3Mo) that caused the increased of tensile and hardness.



a- Metastable b- Equilibrium

Figure 1 phase relationships in the Iron – Nickel system [1]

Table 1 Nominal compositions of commercial Maraging steel according to ASTM designation A 538-65 as specified INCO [3,4 and 5]

Grade MPa	Nominal composition weight $\%_{(a)}$					
	Ni	Mo	Co	Ti	Al	Nb
18Ni1400	18	3.3	8.5	0.2	0.1	-
18Ni 1700	18	5	8.5	0.4	0.1	-
18Ni1900	18	5	9	0.7	0.1	-
18Ni2400	18	$4.2_{(b)}$	12.5	1.6	0.1	-
18Ni 1600cast	17	4.6	10	0.3	0.1	-
$12\text{-}5\text{-}3(1260)_{(c)}$	12	3	-	0.2	0.3	-
Cobalt-Free Nickel 1400	18.5	3	-	0.7	0.1	-
Cobalt-Free Nickel 1700	18.5	3	-	1.4	0.1	-
Low- Cobalt-Nickel 1700	18.5	2.6	2	1.2	0.1	0.1
Cobalt-Free Nickel (1900)	18.5	4	-	1.85	0.1	-

Conditions:
(a) All grades are no more than 0.03%C.
(b) Some procedures use a combination of 4.8% Mo and 1.4% Ti nominal.
(c) Contains 5% Cr.

Table 2 Typical mechanical properties of cast 17 Ni 1600 Maraging steel [5]

Property		Solution annealed		Maraged			
		a	b	c		d	
%0.2 proof stressN/mm2		740	750	1650			
Tensile strength N/mm2		960	990	1730			
Elongation Lo=5d		12	13	7			
Reduction of area%		58	62	35			
Notched/smooth tensile ratio		-	-	1.26			
Modulus of elasticity GN/m2		-	-	188			
Modulus of rigidity GN/m2		-	-	72			
Poissons ratio		-	-	0.3			
Hardness Hv		295	320	530			
Hardness HRC		29	32	49			
Plane strain fracture MNm-2/3		-	-	82.5			
Charpy V-notch impact value at				J	daJ/cm2	J	daJ/cm2
=	-196C			3	0.38	8	1
=	-100C			9	1.1	18	2.3
=	-40C			16	2	20	2.5
=	20C			18	2.3	2.2	2.8

Condition:
Heat treatment: a-Homogenized 4hr at1150^{o}C air cooled
b-4hr at1150^{o}C+4hr at595^{o}C+1hr at820^{o}C air cooled
c-4hr at1150^{o}C+3hr at480^{o}C
d-4hr at1150 ^{o}C+4hr at595^{o}C+1hr at820 ^{o}C+hr at480^{o}C

Table 3 Tensile properties of 16% Ni cast alloy after various heat treatments

Heat treatment temperature ^{o}C+ time hr	Y.S Mpa	U.T.S Mpa	E% In50mm	R%
A825+1hr	570	840	14	55
A1250+1hr	618	910	15	46
650+1hr and G3hr+485	900	1038	20	43
A750+1hr and G3hr +485	1364	1428	8	35
A825+1hr and G3hr+485	1434	1488	9	38
A875+1hr and G3hr+485	1350	1416	8	33
A925+1hr and G3hr+485	1356	1416	8	35
A985+1h and G3hr+485	1368	1438	8	35
A1150+1hr and G3hr+485	1332	1410	8	35
A1250+1hr and G3hr+485	1330	1392	6	24

A-annealing temperature
G-aging temperature

Table 4 Effect of over heating on specimens initially annealing 1hr at 825^{o} C and aged 3hr at 485^{o}C

Over heat temperature ^{o}C (1)	Time min	Vicker Hardness	%γ (2)	Vicker hardness * (3)
545	5	480	3	491
	30	476	6	487
600	5	458	8	500
	30	431	35	497
650	5	375	35	494
	30	345	25	490
750	5	335	8	497
	30	325	7	492

1-one specimen used for each heat treatment.
2-determined by x-ray diffraction.
3-the second annealed and aged were 1hr at 825^{o}C and followed by 3hr at 485^{o}C.
*Vickers hardness after second annealed and aged 3hr .

Figure 2 sample of 16 % Ni cast steel produced by sand casting

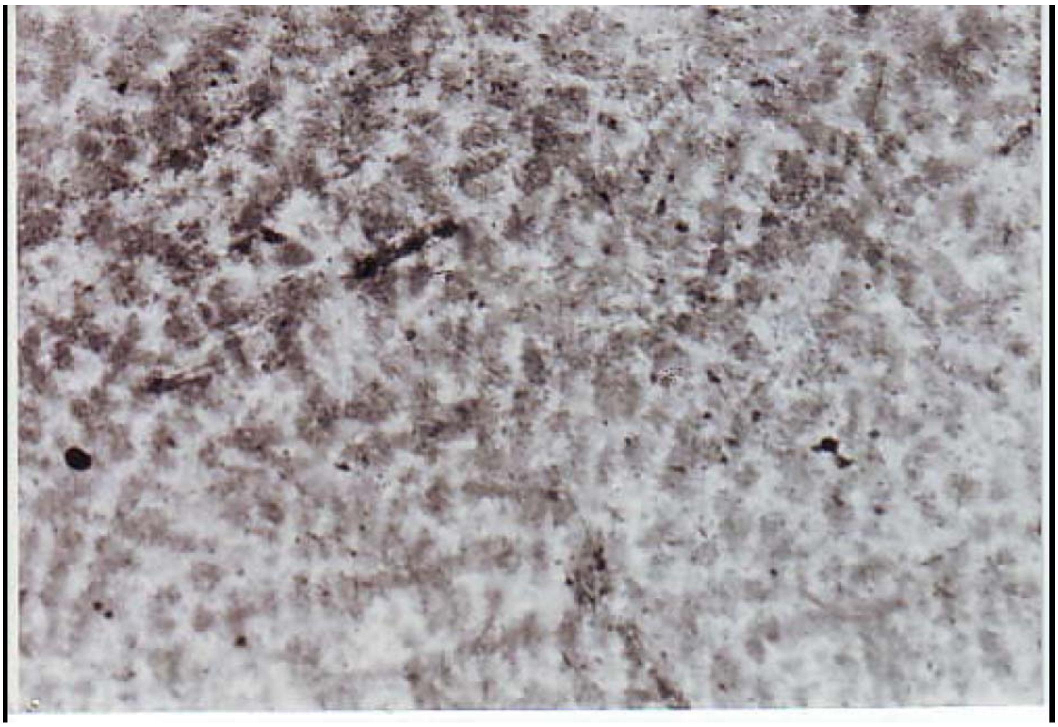


Fig.3. Microstructure produced by annealing 1 hr at 650C .Austenite phase plus bcc phase.Etchant 50ccHcl,25cc HNO3,1g CuCl2,150cc H2o X200

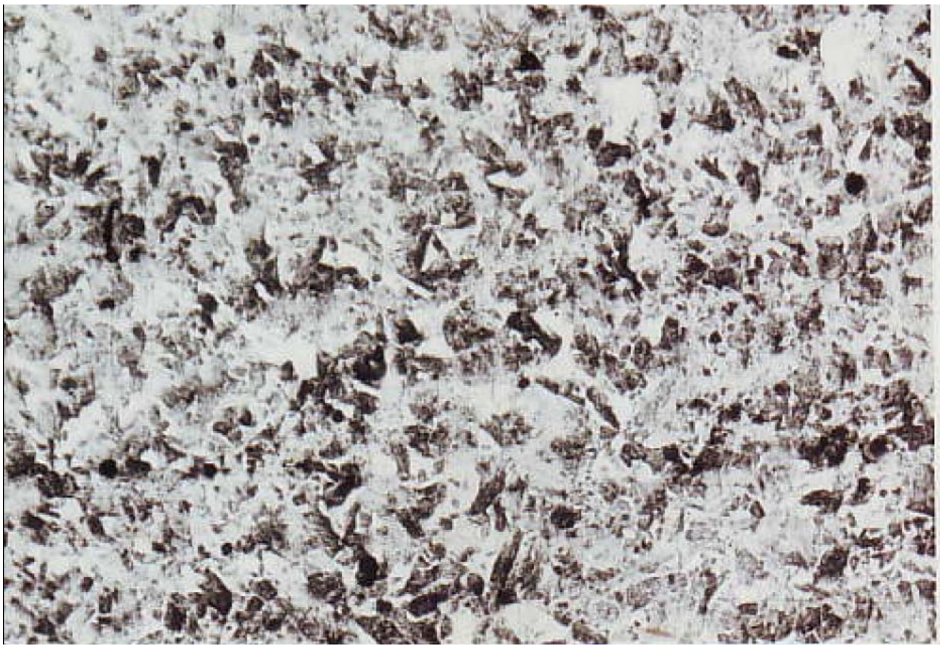


Fig.4.Microstructure produced by annealing 1 hr at 825C.100% bcc phase. Etchant 50ccHcl,25cc HNO3,1g CuCl2,150cc H2o X200.

Fig.5.Microstructure produced by annealing 1 hr at 1250C.100% bcc phase. Etchant 50ccHcl,25cc HNO3,1g CuCl2,150cc H2o X200.

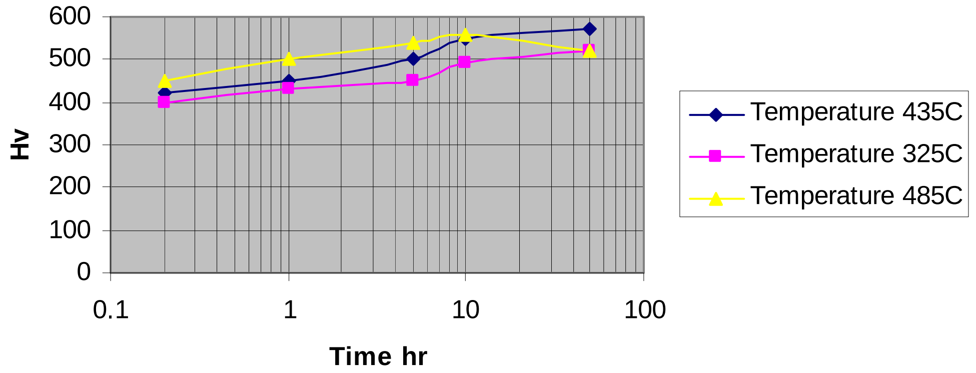


Fig.6.Effect of Aging Temoerature on Hardness of 16%Ni Cast Steel(Initial annealed at 825C)

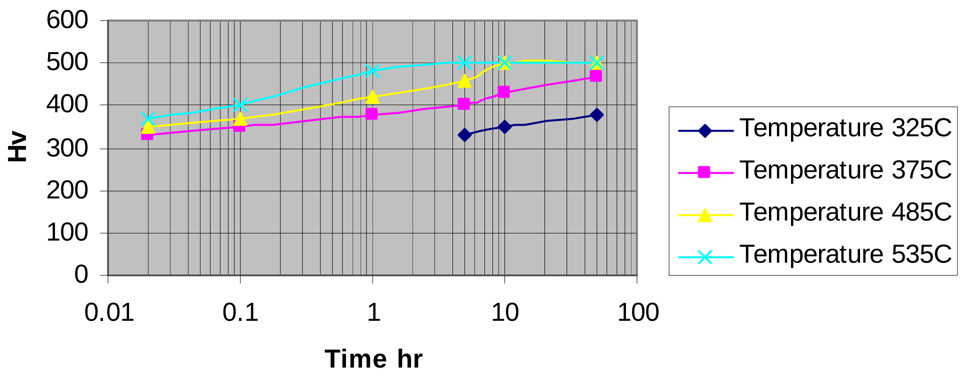


Fig.7.Effect of Aging Temperature on Hardness of 16%Ni Cast Steel(Initial annealed at 985C)

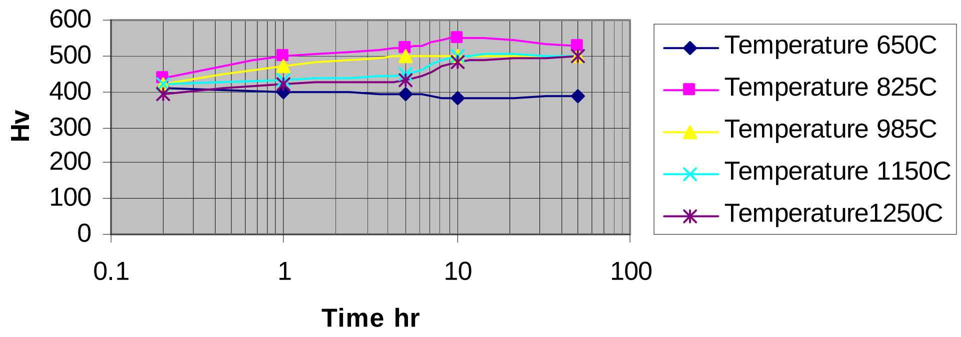


Fig.8. Effect of Aging Temperature at 485C on Hardness of 16%Ni Cast Alloy .(Initial annealed at various temperature)

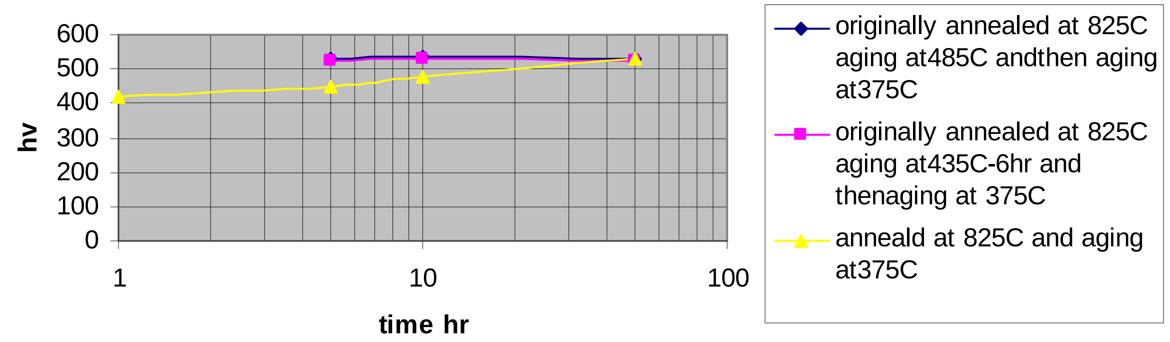


Fig.9.Duble aging result compared of hardness obtained at 375C by single aging with those obtained aftter preliminary aging at high temperature

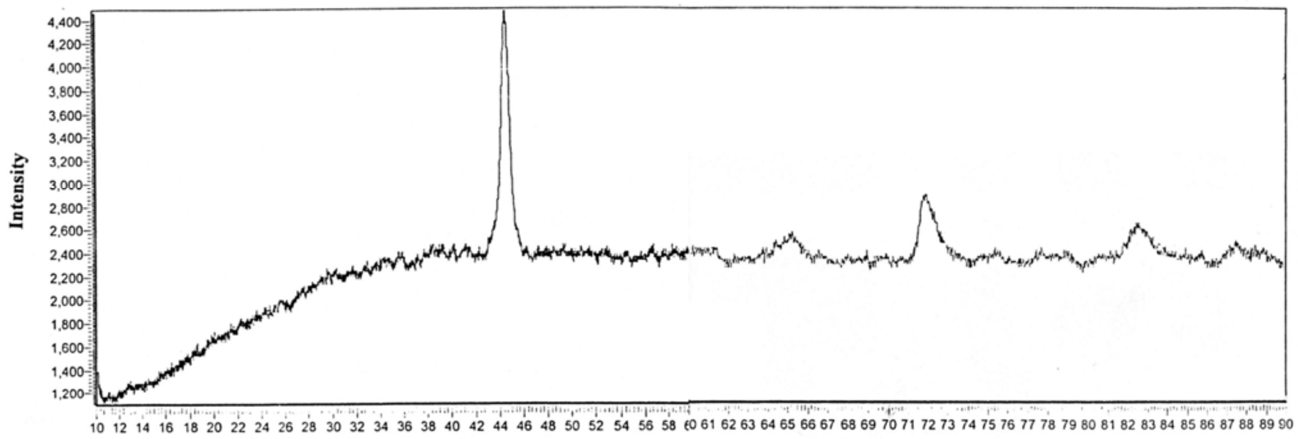


Fig (10) . X-Ray diffraction analysis of specimens after aging at 485° C for 2hr

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Introduction

The discover of wrought 18%Ni Maraging steel has provided class of alloys possessing high strengths and toughness [1].These steels contain 18%Ni and 7-9%Co,3-5%Mo,0.2-0.8%Ti and less than 0.03%C max . They are austenitic at elevated temperature and transform to martensit on cooling to room temperature. The transformation characteristics illustrated in Fig.(1) are form by diagramly Jones and Pumphery[2].Within the limits of martensite in body centered cubic. Reheating the steel to 480 ^{o}C the Maraging treatment produced 1400-1900 Mps yield strength, notch tensile strength/tensile strength ratio over 1.4 and up to 60% reduction in area.

It was believed that the industrial needs for high strength cast alloy with high toughness could be filled by cast Maraging steels .Therefore the more studies included cast Maraging steel.The 17%Ni 1600 grad developed for cast has slighting different composition compared with wrought grades in table(1)[3,4,5]. The 17%Ni 1600 cast Maraging steel grade of nickel Maraging is normally annealed by homogenizing for 4hour at 1150 followed by air cooling. Maraging of the solution annealing material is effected by heating 3 hours at 480C$^{\circ}$ [6, 7].Typical mechanical properties after these treatments are presented in table (2) [6]. The more studies included Maraging steel. Some studied on heat treatment of wrought Maraging steel included ,Floreen and R.F.Decker study the effect of heat treatment and thermo-mechanical treatment of 18%Ni wrought Maraging steel in 1979[8]. Stiller and Daniox study concerned evolution of Mo –rich precipitates of wrought Maraging steel (IRK91) with composition 12%Cr,4%Mo,9%Ni and 2%Cu was investigated after heat treatments at(475 ^{o}C)up to400 hr using atom probe field. The investigation reveled that nucleation of a Mo-rich phase stars between 1 and 2hr of aging at matrix near the Ni- rich precipitates that were found earlier and at the martensite boundaries[9]. The aging behavior of 18Ni(350) maraging steel was studied by Mossbauer spectroscopy. The aging reactions start with the redistribution of atoms, which results in the formation of Fe-Co-rich zones and Ni-Mo-Ti-rich zones at the early stages of aging. The redistribution of atoms is fast at the initial stages of aging and then is slow. The shape of the concentration fluctuation during the early stages of aging is close to a pulse wave. Intermetallic compounds precipitate on the Ni-Mo-Ti-rich zones.[10] . The evolution of precipitates in maraging steel of grade 350 was studied using the complementary techniques of small angle X-ray scattering (SAXS) and transmission electron microscopy (TEM). These investigations revealed that ageing the steel at 450 ^{o}C involved a rhombohedral distortion of the supersaturated b.c.c. martensite accompanied by the appearance of diffuse omega-like structures.

This was followed by the appearance of well-defined omega particles containing chemical order. At the ageing temperature of 510 ^{o}C, Ni-3(Ti, Mo) precipitates were the first to appear with a growth exponent of 1/3. [11].

The age hardening kinetic in temperature range of 713 to 813 K a 2400 Mpa grade coblet- free maraging steel (Fe.18.8-19.1%Ni ,4.4-5.4%Mo,2.6%Ti)has been studied by YIHE, KE YANG and others. The study included microstricture and mechanical properties that showed a high number of(Ni3Ti) and (Fe2(Mo,Ti)) precipitates were formed during the aging process[12]. The effect of the heat treatment included solution treatment and aging treatment on the proire austenite grain size microstrcture and mechanical properties of a precipitates hardening maraging stainless steel was investigation by KAILIV,YIYIN SHAN and others [13].

Experimental procedure

The composition of the cast alloys studied was the follows 69.77%Fe, 0.02%C,16.34%Ni,4.85%Mo,8.65%Co,0.6%Ti,0.13%Al,0.12%Cr,0.39%V and traces contains of Zr,Cu,Si and Mn.

The alloy was mad as a (4Kgs) charge that melted using a high frequency rapid induction melting furnace supple as high as (60 Kw) at nominal frequency of (19.6 KHz)from (415-440 V). A standard melt super pouring temperature of (1600±10 ^oC) was maintained to achieved a pouring temperature (1500±10).The material used in the present study was taken from product of sand casting as solid bar shape with dimensions(Θ25*200mmlength) that shown in Fig(2). The solid bar has homogenization treatment at 1150^{o}C for one hour and air cooling to room temperature. The alloys were studied by Vickers hardness measurement augmented by tensile test and structured studies. Preliminary work had shown that the hardness values correlated quit well with yield strength. Thus the hardness measurement furnished a simple and reasonably reliable method of evaluating the mechanical properties.

The effect of annealing temperature on tensile strength were studied on samples which cutting as bar according DIN 50125-As*25 and annealed for 1 hr at temperature range from 650 to 1250 ^{o}C. The aging behavior of the alloy after these various initial treatments were then evaluated by heat treated the samples in the temperature range of 325 to 545 ^{o}C for times ranging from half min to several hours.

Most of the heat treatments were done in nitrogen atmosphere in vacuum furnace except for that short time aging tests which were done in salt bath to insure rapid heating. All of the specimens were gas cooled as prior tests had indicated no effects due to cooling rate. A number of light examination and X-ray diffraction analyses also used.

Results and Discussion

Annealing Behavior

In this project the annealing treatment occur on cast alloy structure at different temperature.

When the cast alloy was annealed at 650 ^{o}C the structure contained approximately35% austenite after cooling to room temperature. This austenite had the fine, lamellar appearing structure as shown in Fig. (3). Apparently at this annealing temperature the austenite that found during annealing became partially stabilized and did not retransform to body-centered cubic structure on cooling to room temperature. This retained austenite may have been due to transformation of the metastable body centered cubic martensite($\alpha 2$) matrix found on cooling into the equilibrium ferrite (α)and austenite(γ) phase asX-ray diffraction shown. During this transformation there was probably some partitioning of the alloying elements between the α and γ phases so that some of the austenite was enriched and did not transform back to $\alpha 2$ on cooling to room temperature.

When the annealing temperature was raised the amount of austenite retained on cooled to room temperature decreased until only the body-centered cubic phase was present. The minimum annealing temperature required to eliminate all the austenite was approximate 750 ^{o}C.At temperature 650 ^{o}C and below. When reheated a cast sample for this temperature for a period long enough to establish equilibrium ferrite of composition α and austenite of the composition (γ) are formed from the previously existing martensite. It is seen that the ferrite is leaner in nickel and the austenite is richer in nickel that the prior martensite. On cooling back to room temperature the ferrite does not transform to martensite. In addition the austenite may be sufficiently enriched with nickel that it becomes stable and therefore does not transform to martensite. To return the austenite to transformation condition. It necessary to reheat to high temperature more than 650 °C that lie the materials in the single phase gamma region where the austenite can be restored to it original composition.

When aging this above specimens the result appears eliminate this austenite to achieve satisfactory hardening during aging. The martensite is perfect at aging after annealing temperature 825 °C the microstructure in Fig. (4) Appear this structure. The austenite grain size was No 6 to 8 according the ASTM after annealing at temperature to 980 °C. Above this temperature grain growth accorded with ASTM grain size No 0 after at 1250 °C.

With high annealing temperatures a widmansttaten morphology was evident in the microstructures of the body centered cubic $\alpha 2$ phase as in Fig.(5) while as explain in above the annealing at 825 °C appear the microstructure have100% bcc with low amount of widmanstatten structure. As the structure in Fig.(4),(5) were entirely body centered cubic the X-ray diffraction shown no differences in chemical composition between the dark plates and light back ground in area. Also the micro hardness appears no different between two areas in Fig.(5). The tensile properties after annealing at 825 °C to 1250 °C are listed in table 3 also the tensile properties obtained after various heat treatments are included in table(3). The general findings were in accord with the hardness-1hr at 825 °C gave yield strength of 1434Mpa.Raising the annealing temperature to 875 °C lowered the annealing 1350 °C. Further increase of the annealing temperature had little effect on strength.

Aging behavior

The hardness of the specimens initially annealed at 825 °C and then aging are shown in Fig .(6). More extensive data for specimens initially annealed at 985 °C are shown in Fig. (7). The curves show a rapid initial rate of hardening at 485 to 545°C while the response at 325 to 375°C was slower. The general shapes of these curves are comparable to these of precipitation hardenable alloys. In the present alloy, however over aging is the conventional sense may not occur because have short time for aging reached to 48 hrs. The think that the hardness will drop with prolonged aging because that reformation of austenite .The general appearance of the austenite form after prolonged aging was similar to the fine lamellar type of the in Fig.(3). The amount of austenite that was observed after any given aging treatment varied somewhat with the initial conditions. Prior amount of austenite in higher annealing temperatures reduced the austenite content.

The effects of various initial annealing treatments on the hardness after aging at 485°C are shown in Fig. (8). Changing the initial condition of the alloy prior to aging produced some distinct changes in hardness behavior. The specimens that annealed at 650°C contained about 35% austenite and were not hardening by the aging treatment. Raising the annealing temperature from 825 to 1250 °C resulted in lower hardness after aging.

There is still broad field in annealing and aging conditions to yield reproducible high strengths. First the stabilization of austenite by annealing below 735 ^{o}C or by aging to high temperature should be avoided to obtain optimum strength.

There also the aging treatment have considerable field because of the relatively flat nature of the hard time curve at 485 ^{o}C. For most purposes aging for 3hr at 485 ^{o}C would be the normal heat treatment but varying the aging time at 485 ^{o}C from 2 to 6hr does not significantly alter the hardness. Aging could also be done at other temperature. But the increase in the rate of revelation to austenite at higher temperatures and sluggishness of reaction at lower temperatures. Indicated that 485 ^{o}C ±10 is probably the optimum aging temperature for cast alloy. Strength can be lowered by higher annealing temperature and or aging at lower temperatures. For application requiring elevated temperature strength the use of higher annealing temperatures is advantageous in retarding austenite reversion and thus maintaining the strength properties for longer time periods. Some gain in strength was also noted by aging at435 ^{o}C for 24hr.

The X-ray diffraction evident indicated as in Fig (10) the strength occurring above alloys on aging at 480 ^{o}C results from the early formation of zones or clusters based on an Ni3Mo grouping containing iron as (Ni,Fe)3Mo which at higher aging temperatures may give way or evolve into a precipitate of Fe2Mo . At the lower aging temperatures and long holding times the clusters may perhaps be supplemented by the Fe2Mo precipitate. The third precipitate containing titanium forms in the promotion of age hardening in these cast steels this precipitate is FeTi sigma phase.

Double Aging

Several experiments were conducted to study the effects of double aging heat treatment specimens were initially aging at one temperature and then aging for various times at second temperature. The result showed that when the specimens were aging at lower temperature and then at 485 ^{o}C the resultant hardness curves at 485 ^{o}C closely followed the normal 485 ^{o}C aging curve .Of more interest are the results in Fig.(9) showing that when specimens were first aging at 435 or485 ^{o}C to produced about 530 Vickers hardening and then held at 375C ^{o}C there was essentially little change in hardness after 48hr at 375 ^{o}C. The curves in fact crossed that obtained as a result of aging only at 435 ^{o}C from above results excluded the results indicated that there is no special advantage to be gained by double aging treatments. One possible benefit of the apparent stability of the aging structure at lower aging temperatures might occur in elevated temperature service applications in the 375 ^{o}C. In such cases the alloy when aging at 485 ^{o}C might not be susceptible to the long-time hardening and embitterment that occur in many alloys.

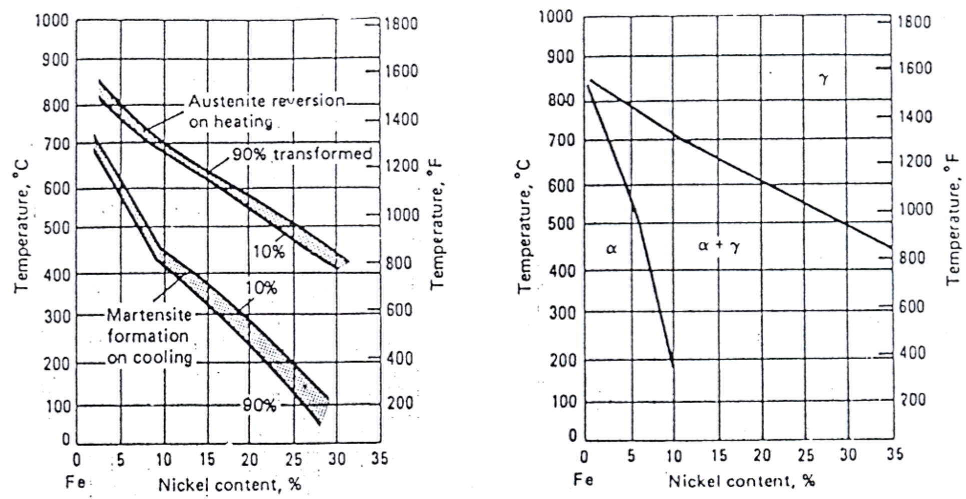
Over Heat after Aging

When the cast alloys samples are heated for extended period of time at higher aging temperature the over aged of matrix tends to revert to austenite. The same thing happens on heating at temperatures between the aging range and the annealing range. The results of these tests listed in table(4) indicated that the original hardness was essentially restored by re-annealing and re-aging. With over heating there was a progressive decrease in hardness with increasing time and temperature. The extent of the reversion depends on the time and temperature, thus the aging 10hr at 485 ^{o}C which they considered to started of reverted austenite. Greater amounts of the phase were seen in material aged 48hr at the same temperature. The X-ray diffraction study indicated the presence of approximate 12 to 14% austenite in these specimens. 3% austenite was found in cast steel aged 6 minutes at 545 °C, where as 6% austenite was developed by extending the age time to 30 minute. We thinks the amount of austenite increased and then deceased with increasing temperatures probably because of changes in stabilization of the austenite thus at the lower overheating temperature

The austenite that formed was probably due to the α2 to α+ (γ) reaction and was sufficiently enriched in alloy content so that it did not transform back on cooling. At higher over heating temperatures the austenite probably formed by the α2 to (γ) reaction and most of the austenite transformed back to α2 on cooling to room temperature. However when the specimen were re-annealed and re-aged the hardness returned substantiating to the average initial hardness before over heating.

Conclusions

1- The annealing temperature of 16%Ni cast Maraging steels must be occur in range 825±10 °C and the rang of 600 to 700 °C must be avoided to prevent retained austenite.
2- Marked hardening during aging occurs in several minutes at 485C or 545 °C. and optimum treatment occur after 3-6 hr at 485 ± 10 °C.
3- Larger amounts of retained austenite are present after over aging that appear after 5 to 30 minute in range 545 to750 °C.The initial hardening can be restored by re-annealing and re-aging.
4- The results of double aging appear that the aging at lower temperature followed by a second aging at 485 °C produces hardness value equal to values produced by a single aging at 485 °C and the specimens first aged at 485 °C or 435 °C and then aged at 375 °C no change in hardness after 48 hr.
5- Electron diffraction studies of the aging cast gave evidence of phase (Fe2CoNi and Ni3Ti, Ni3Mo) that caused the increased of tensile and hardness.



a- Metastable b- Equilibrium

Figure 1 phase relationships in the Iron – Nickel system [1]

Table 1 Nominal compositions of commercial Maraging steel according to ASTM designation A 538-65 as specified INCO [3,4 and 5]

Grade MPa	Nominal composition weight %$_{(a)}$					
	Ni	Mo	Co	Ti	Al	Nb
18Ni1400	18	3.3	8.5	0.2	0.1	-
18Ni 1700	18	5	8.5	0.4	0.1	-
18Ni1900	18	5	9	0.7	0.1	-
18Ni2400	18	4.2$_{(b)}$	12.5	1.6	0.1	-
18Ni 1600cast	17	4.6	10	0.3	0.1	-
12-5-3(1260)$_{(c)}$	12	3	-	0.2	0.3	-
Cobalt-Free Nickel 1400	18.5	3	-	0.7	0.1	-
Cobalt-Free Nickel 1700	18.5	3	-	1.4	0.1	-
Low- Cobalt-Nickel 1700	18.5	2.6	2	1.2	0.1	0.1
Cobalt-Free Nickel (1900)	18.5	4	-	1.85	0.1	-

Conditions:

(a) All grades are no more than 0.03%C.
(b) Some procedures use a combination of 4.8% Mo and 1.4% Ti nominal.
(c) Contains 5% Cr.

Table 2 Typical mechanical properties of cast 17 Ni 1600 Maraging steel [5]

Property		Solution annealed		Maraged			
		a	b	c		d	
%0.2 proof stressN/mm2		740	750	1650			
Tensile strength N/mm2		960	990	1730			
Elongation Lo=5d		12	13	7			
Reduction of area%		58	62	35			
Notched/smooth tensile ratio		-	-	1.26			
Modulus of elasticity GN/m2		-	-	188			
Modulus of rigidity GN/m2		-	-	72			
Poissons ratio		-	-	0.3			
Hardness Hv		295	320	530			
Hardness HRC		29	32	49			
Plane strain fracture MNm-2/3		-	-	82.5			
Charpy V-notch impact value at				J	daJ/cm2	J	daJ/cm2
=	-196C			3	0.38	8	1
=	-100C			9	1.1	18	2.3
=	-40C			16	2	20	2.5
=	20C			18	2.3	2.2	2.8

Condition:

Heat treatment: a-Homogenized 4hr at1150^{o}C air cooled
b-4hr at1150^{o}C+4hr at595^{o}C+1hr at820^{o}C air cooled
c-4hr at1150^{o}C+3hr at480^{o}C
d-4hr at1150 ^{o}C+4hr at595^{o}C+1hr at820 ^{o}C+hr at480^{o}C

Table 3 Tensile properties of 16% Ni cast alloy after various heat treatments

Heat treatment temperature ^oC+ time hr	Y.S Mpa	U.T.S Mpa	E% In50mm	R%
A825+1hr	570	840	14	55
A1250+1hr	618	910	15	46
650+1hr and G3hr+485	900	1038	20	43
A750+1hr and G3hr +485	1364	1428	8	35
A825+1hr and G3hr+485	1434	1488	9	38
A875+1hr and G3hr+485	1350	1416	8	33
A925+1hr and G3hr+485	1356	1416	8	35
A985+1h and G3hr+485	1368	1438	8	35
A1150+1hr and G3hr+485	1332	1410	8	35
A1250+1hr and G3hr+485	1330	1392	6	24

A-annealing temperature
G-aging temperature

Table 4 Effect of over heating on specimens initially annealing 1hr at 825^o C and aged 3hr at 485^{o}C

Over heat temperature ^oC (1)	Time min	Vicker Hardness	%γ (2)	Vicker hardness * (3)
545	5	480	3	491
	30	476	6	487
600	5	458	8	500
	30	431	35	497
650	5	375	35	494
	30	345	25	490
750	5	335	8	497
	30	325	7	492

1-one specimen used for each heat treatment.
2-determined by x-ray diffraction.
3-the second annealed and aged were 1hr at 825^{o}C and followed by 3hr at 485^{o}C.
*Vickers hardness after second annealed and aged 3hr .

Figure 2 sample of 16 % Ni cast steel produced by sand casting

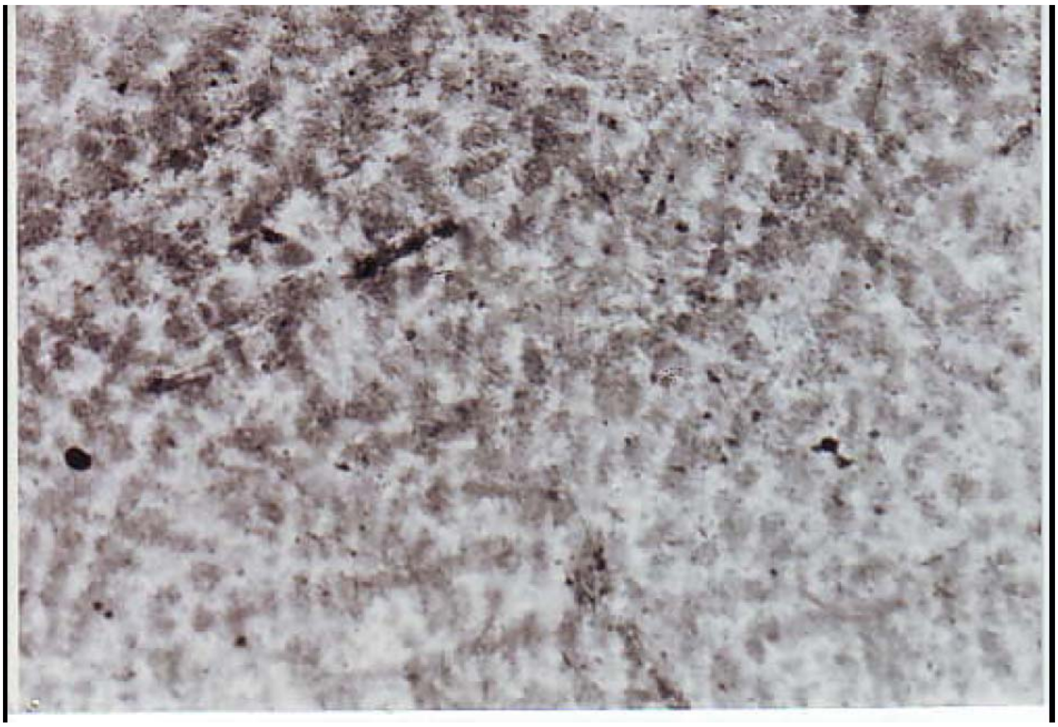


Fig.3. Microstructure produced by annealing 1 hr at 650C .Austenite phase plus bcc phase.Etchant 50ccHcl,25cc HNO3,1g CuCl2,150cc H2o X200

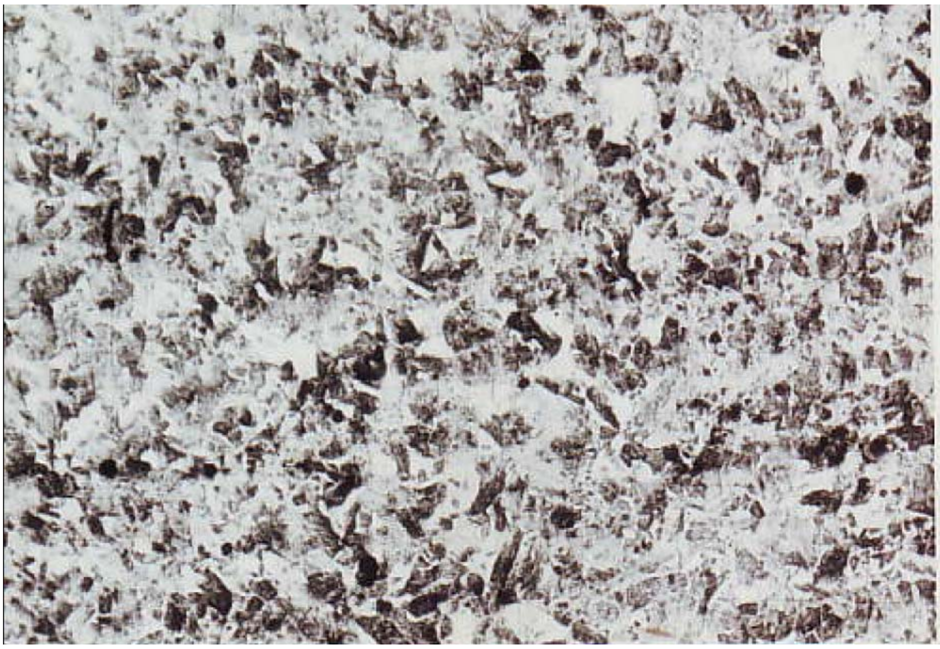


Fig.4.Microstructure produced by annealing 1 hr at 825C.100% bcc phase. Etchant 50ccHcl,25cc HNO3,1g CuCl2,150cc H2o X200.

Fig.5.Microstructure produced by annealing 1 hr at 1250C.100% bcc phase. Etchant 50ccHcl,25cc HNO3,1g CuCl2,150cc H2o X200.

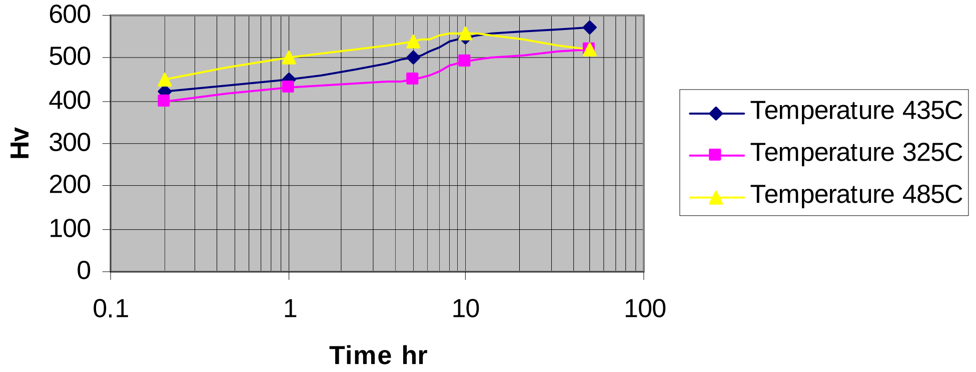


Fig.6.Effect of Aging Temoerature on Hardness of 16%Ni Cast Steel(Initial annealed at 825C)

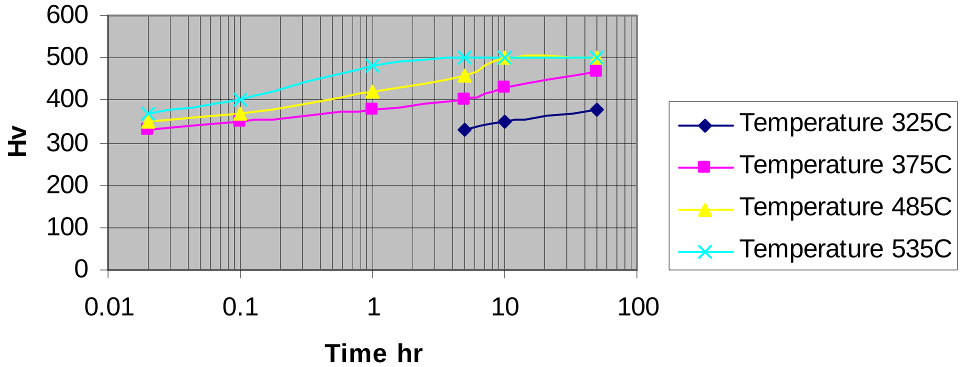


Fig.7.Effect of Aging Temperature on Hardness of 16%Ni Cast Steel(Initial annealed at 985C)

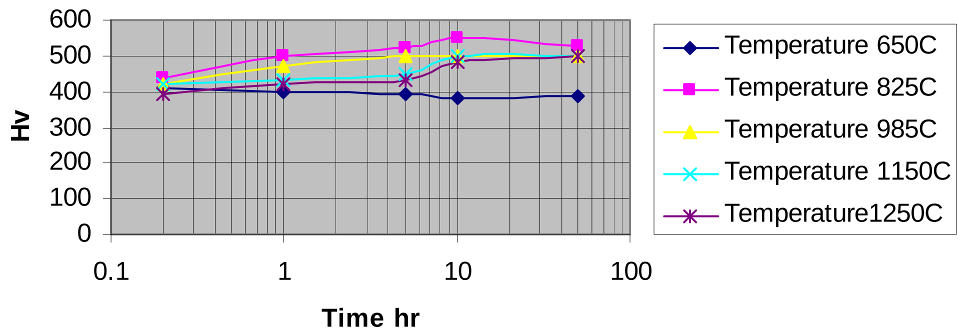


Fig.8. Effect of Aging Temperature at 485C on Hardness of 16%Ni Cast Alloy .(Initial annealed at various temperature)

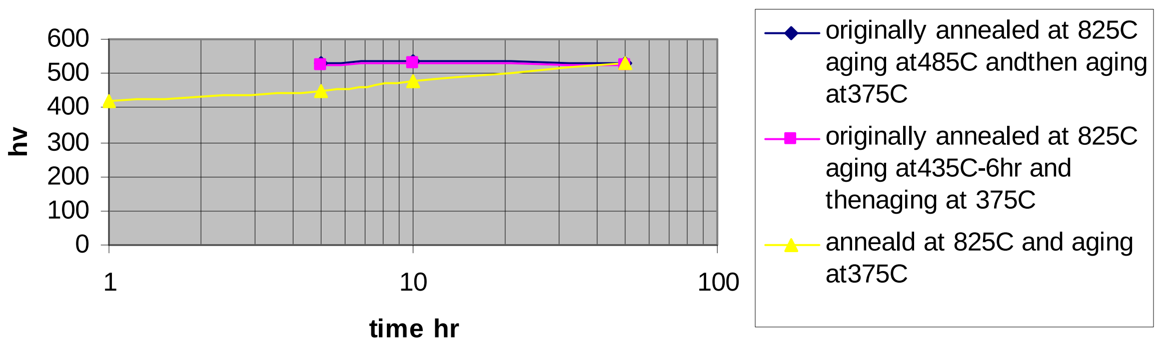


Fig.9.Duble aging result compared of hardness obtained at 375C by single aging with those obtained aftter preliminary aging at high temperature

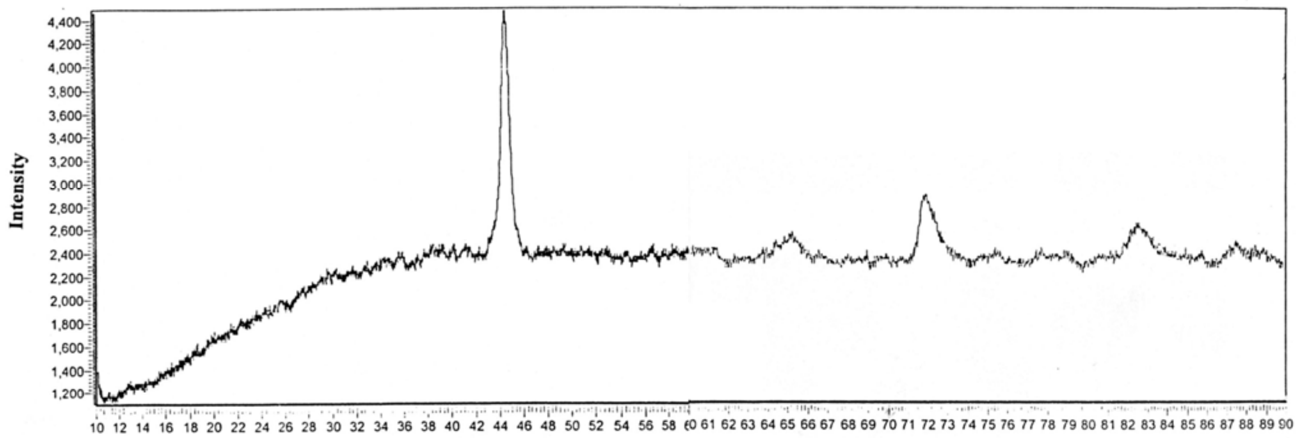


Fig (10) . X-Ray diffraction analysis of specimens after aging at 485° C for 2hr

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