

AD-758 439

Processing and Properties of 18Ni Maraging Steel by Powder Metallurgy

Army Materials and Mechanics Research Center

FEBRUARY 1973

Distributed By:

NTIS

**National Technical Information Service
U. S. DEPARTMENT OF COMMERCE**

AMMRC TR 73-4

AD

AD 758439

PROCESSING AND PROPERTIES
OF 18Ni MARAGING STEEL
BY POWDER METALLURGY

ERNEST P. ABRAHAMSON, II
MATERIALS APPLICATION DIVISION

February 1973

NATIONAL TECHNICAL
INFORMATION SERVICE

DDC
RECEIVED
APR 17 1973
RECEIVED
C

Approved for public release; distribution unlimited.

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

16

ACCESSION for	
NTIS	White Section <input type="checkbox"/>
906	Buff Section <input type="checkbox"/>
UNANNOUNCED	<input type="checkbox"/>
JUSTIFICATION	
BY	
DISTRIBUTION/AVAILABILITY CODES	
Dist.	APIL 2 / 10 1964
A	

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

Mention of any trade names or manufacturers in this report shall not be construed as advertising nor as an official indorsement or approval of such products or companies by the United States Government.

DISPOSITION INSTRUCTIONS

Destroy this report when it is no longer needed.
Do not return it to the originator.

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Army Materials and Mechanics Research Center Watertown, Massachusetts 02172		2a. REPORT SECURITY CLASSIFICATION Unclassified	
		2b. GROUP	
3. REPORT TITLE PROCESSING AND PROPERTIES OF 18Ni MARAGING STEEL BY POWDER METALLURGY			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)			
5. AUTHOR(S) (First name, middle initial, last name) Ernest P. Abrahamson, II			
6. REPORT DATE February 1973		7a. TOTAL NO. OF PAGES 14	7b. NO. OF REFS 9
8a. CONTRACT OR GRANT NO.		9a. ORIGINATOR'S REPORT NUMBER(S) AMMRC TR 73-4	
b. PROJECT NO. D/A 1B564603D66300			
c. AMCMS Code 554C12.26300-X032384		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
d.			
10. DISTRIBUTION STATEMENT Approved for public release; distribution unlimited.			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Picatinny Arsenal Dover, New Jersey 07801	
13. ABSTRACT Using 18Ni Mar 300 powder processed by the rotating electrode process, rods have been extruded under varying temperatures and ram speeds. It is observed that control of both variables allows structure control and increases the Charpy V-notch toughness from 7 to 23 ft-lb at room temperature. (Author)			

DD FORM 1473

REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

UNCLASSIFIED
Security Classification

14

KEY WORDS

LINK A

LINK B

LINK C

ROLE

WT

ROLE

WT

ROLE

WT

Maraging steel
Mechanical properties
Metallography
Powder metallurgy
Extruding

AMMRC TR 73-4

PROCESSING AND PROPERTIES OF 18Ni MARAGING STEEL BY POWDER METALLURGY

Technical Report by
ERNEST P. ADRAHAMSON, II

February 1973

D/A Project 1B564603D66300
AMCMS Code 554C.12.26300-XO32384
Projectile, 8" XM673

Approved for public release; distribution unlimited.

**MATERIALS APPLICATION DIVISION
ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172**

CONTENTS

	Page
ABSTRACT	
INTRODUCTION	1
PROCEDURE	1
MICROSTRUCTURE	2
MECHANICAL PROPERTIES	6
DISCUSSION	8
CONCLUSIONS	9
ACKNOWLEDGMENT	9
LITERATURE CITED	10

INTRODUCTION

The alloy compositions of the commercial 18% Ni-Mo-Co maraging steels were designed to provide maximum toughness at strength levels of 200 to 350 ksi. They owe their unique properties to a tough ductile iron-nickel martensite structure which age hardens at approximately 900 F.¹ The primary precipitate responsible for the strengthening is Ni₃Mo.²⁻⁴ Cobalt enhances the effect of Ni₃Mo by decreasing the solubility of molybdenum in the matrix.^{5,6} Ni₃Ti has been identified in maraging alloys, but is believed to be a secondary precipitation hardening reaction.^{4,7,8}

Strength in these materials is increased by raising the titanium content in base compositions that are progressively slightly enriched in nickel, cobalt, and molybdenum to maintain the optimum toughness. At and above 300 ksi yield strength the enriched compositions are subject to austenite retention at regions of alloy segregation.

This report deals with using rotating-electrode-processed powder in an effort to reduce segregation and improve properties.

PROCEDURE

Commercial 300 18% nickel maraging steel was converted to spherical powders using the rotating electrode method. The composition of the alloy was as follows: 0.012 C, 0.05 Si, 0.07 Mn, 0.006 S, 0.004 P, 4.80 Mo, 8.80 Co, 18.05 Ni, 0.05 Ca, 0.10 Al, 0.70 Ti, 0.003 B, 0.009 Zr. The powders were divided into four equal weights and blended in a V-cone blender prior to being placed in the extrusion can, evacuated, and sealed.

All billets were heated at 1500 F for 3 hours and compacted under 900 tons pressure, ejected, quenched, and the extrusion can remachined.

The four billets were extruded with a ten-to-one reduction followed by a water quench. The temperatures and other extrusion data are shown in Table I.

Table I. EXTRUSION DATA

Billet	Extrusion Temperature, deg F	Ram Speed, in./min	Extrusion Force, Tons		Extrusion Constant psi	
			Upset	Running	Upset	Running
1	1650	100	825	735	35.7	32.6
2	1400	100	1110	950	47.7	40.8
3	1400	220	1110	965	47.7	41.5
4	1400	400	1130	1000	48.5	43.0

The specimens were tested in three conditions:

Treatment A - as extruded and aged three hours at 900 F

Treatment B - as extruded, solutionized at 1500 F for one hour, water quenched and aged three hours at 900 F

Treatment C - as extruded, solutionized at 1700 F for one hour, water quenched, solutionized at 1500 F for one hour, water quenched and aged three hours at 900 F.

The latter two treatments are those used on conventional wrought 18Ni Mar 300 maraging steel.

All materials were investigated metallographically using both light and transmission electron microscopy. The etch used for light microscopy was 15 cc HCl, 10 cc acetic acid, 10 cc HNO₃, and two drops of glycerin. The specimens for transmission microscopy were thinned using the window method at -40 F in a solution of 30 cc perchloric acid, 175 cc 1-butanol, and 275 cc methanol at 6 volts and 0.05 amperes.

MICROSTRUCTURE

The structure of the rotating-electrode-processed powder is shown in Figure 1. The dendrite arm spacing is approximately 3 microns. Throughout the particle one can see blocky precipitates of TiC and TiN⁹ randomly dispersed.

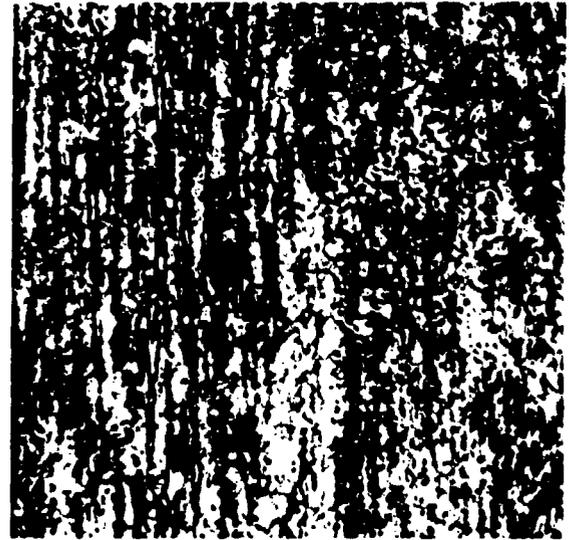
When consolidated and extruded the powder structure is completely lost, see Figure 2. The 1600 F extrusion is recrystallized and the blocky precipitates are still randomly dispersed. However, at 1450 F and 100 in./min the grains are elongated and the precipitate appears in stringers. Additionally, a second precipitate, Ni₃Mo, has started to appear. As the speed of extrusion was increased to 400 in./min at 1450 F the structure becomes a combination of elongated and recrystallized grains, with no indication of Ni₃Mo and a decreasing number of TiN and TiC stringers. This latter observation is emphasized by aging the extruded structure for three hours at 900 F, see Figure 3. In the lower temperature extrusions there is more massive precipitation of Ni₃Mo along the bands of TiN than in the 1600 F extrusion.



Figure 1. Microstructure of 18Ni Mar 300 Rotating-Electrode-Processed Powder. 1000X



a. 1600 F - 100 in./min



b. 1450 F - 100 in./min

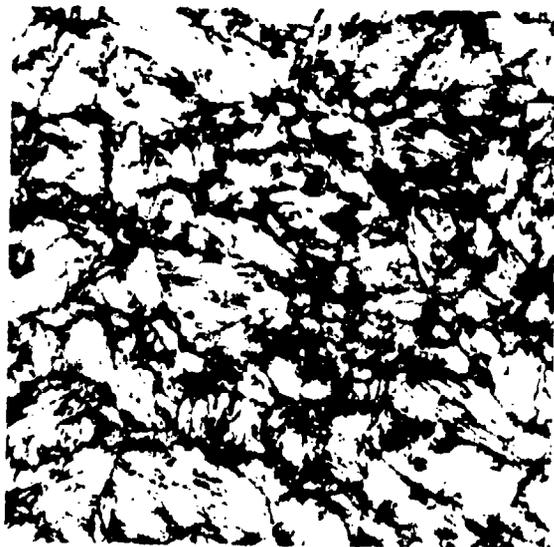


c. 1450 F - 220 in./min



d. 1450 F - 400 in./min

Figure 2. Photomicrographs of As-Extruded 18Ni Mar 300 Steel. Mag. 1000X



a. 1600 F - 100 in./min



b. 1450 F - 100 in./min



c. 1450 F - 220 in./min



d. 1450 F - 400 in./min

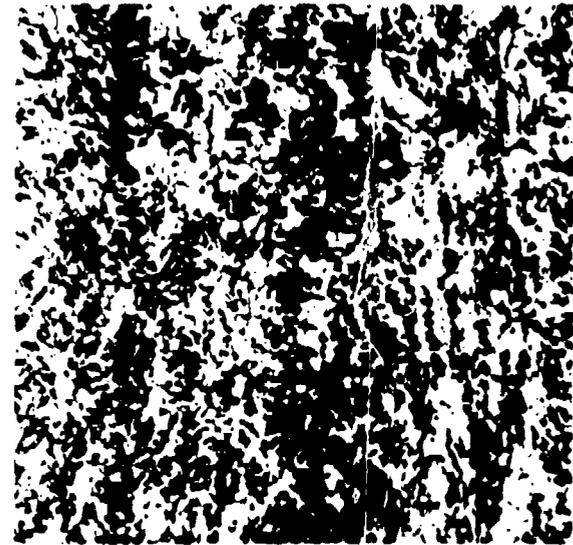
Figure 3. Photomicrographs of 18Ni Mar Steel, As-Extruded and Aged at 900 F for 3 Hr. Mag. 1000X

Three extrusions were selected for solutionizing treatments, deleting that at 1450 F - 220 in./min, which was considered redundant. The structure of the specimens given Treatment B is shown in Figure 4. The prior austenite grain structure of all the extrusions is not as obvious as those specimens that were just aged. The martensite lath structure is considerably refined by the 1500 F solution treatment, see Figure 5. The stringers are still observable in the two 1450 F extrusions, but not quite as obvious in the material extruded at 400 in./min.

Using the higher solutionizing treatment, Treatment C, coarsens the prior austenitic and lath structure, see Figure 6. The indications of fibering are almost obliterated.



a. 1600 F - 100 in./min



b. 1450 F - 100 in./min



c. 1450 F - 400 in./min

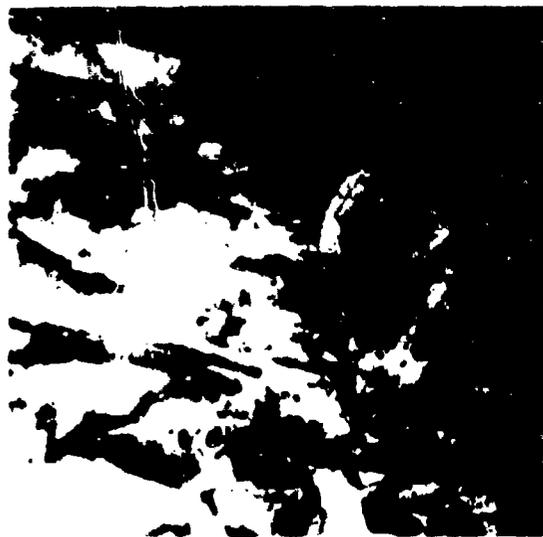
Figure 4. Photomicrograph of 18Ni Mar 300 Steel, As-Extruded, Solutionized at 1500 F for 1 Hr, Water Quenched, Aged at 900 F for 3 Hr. Mag. 500X

MECHANICAL PROPERTIES

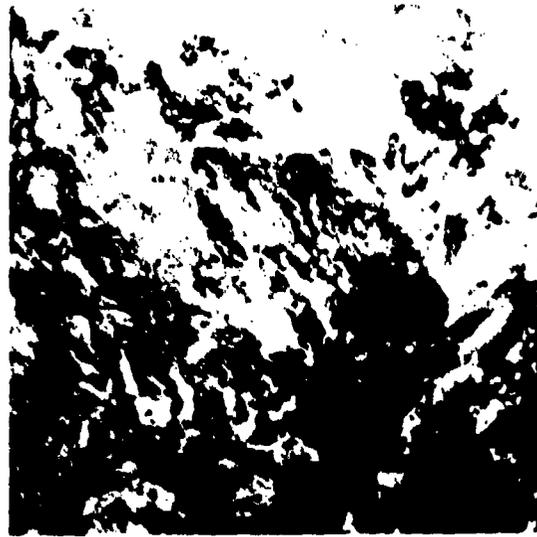
Room temperature tension tests, Charpy V-notch and hardness measurements were run on extrusions 1, 2, and 4. Extrusion 3 was omitted for the same reason noted in the previous section. The as-extruded hardness for all three was R_C 32. The properties for the heat-treated materials and a comparison with commercial material are given in Table II. Within experimental error, the hardnesses are essentially the same for all conditions.

The yield strengths in the aged and 1500 F solution-treated-and-aged conditions are slightly higher than the commercial material (at best 10%). However, the toughness of all three extrusions in these conditions equaled or bettered that of the commercial material. The as-extruded-and-aged material for the 1450 F extrusions nearly doubled the results for commercial 18Ni Mar 300, while the 1600 F extrusion more than tripled the Charpy V-notch value. That material which had an additional 1700 F solution treatment showed the lowest yield and tensile strengths, but showed an improvement in impact values over the 1500 F solution treatment.

The fracture surfaces varied with the extrusion conditions and heat treatment. The material extruded at 1600 F showed a normal fine grain fracture on the Charpy specimens. The lower temperature extrusions had fibrous fractures, with the material extruded at 400 in./min showing a combination of fibrous and fine grain fracture. With an increase in the solution treatment temperature, the fibrosity was seen to decrease.

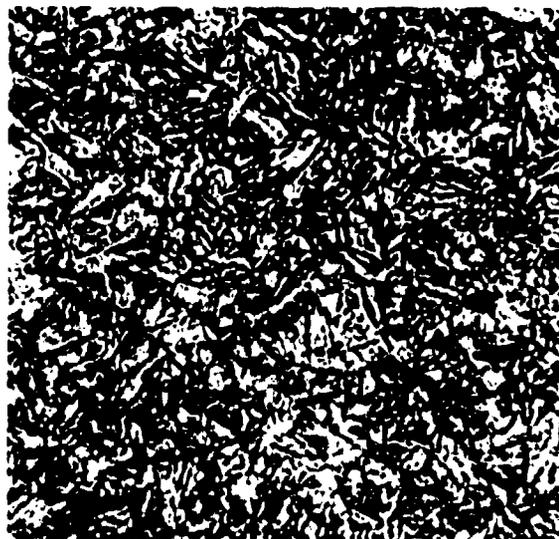


a. As Extruded and Aged at 900 F for 3 Hr



b. As-Extruded at 1500 F for 1 Hr,
Water Quenched and Aged at 900 F for 3 Hr

Figure 5. Transmission Electron Micrograph of 18Ni Mar 300 Steel
Extruded at 1600 F - 100 in./min. Mag. 9000X



a. 1600 F - 100 in./min



b. 1450 F - 100 in./min



c. 1450 F - 400 in./min

Figure 6. Photomicrographs of 18Ni Mar 300 Steel,
As-Extruded, Solutionized 1700 F for 1 Hr, Water Quenched 1500 F for 1 Hr,
Water Quenched and Aged 900 F for 3 Hr. Mag. 500X

DISCUSSION

The processing of the powders by the rotating electrode process has alleviated the problem of alloy segregation which causes "banding" in commercial material. However, the microstructures and properties indicate that care must be taken to control fibering of TiN and TiC particles. Consider the material extruded at 1600 F - 100 in./min, 1450 F - 400 in./min, and 1450 F - 100 in./min as three stages of the fibering sequence.* This indicates that the matrix must be reasonably soft, i.e., at the recrystallization temperature, to avoid fibering.

The best combination of strength and toughness was observed in all cases for the as-extruded and aged condition. Solutionizing at 1500 F plus aging refines the lath martensite structure and has a finer Ni₃Mo precipitate which further increases the strength, but decreases the toughness. Coarsening the structure by higher solutionizing temperatures reduces the strength and increases the toughness. Increasing temperature also decreases the fibering.

Table II. MECHANICAL PROPERTIES FOR 18Ni Mar 300 UNDER VARIOUS CONDITIONS

Material	Heat Treatment	0.1% Yield Stress ksi	0.2% Yield Stress ksi	Ultimate Tensile Stress ksi	True Fracture Stress ksi	Elong %	R. A. %	Charpy V-Notch Room Temp ft-lb	Hardness R _c
Commercial 300 Grade	1500 F-1hr, W.Q., 900 F-3hr, A.C.	265	266	273	381	11.5	40	7	51
REP* 300 Grade Extruded at 1650 F, 100 in./min and Water Quenched	900 F-3hr, A.C.	270	278	282	400	10	54	23	52
	1500 F-1hr, 900 F-3hr, A.C.	282	289	293	357	10	40	11	54
	1700 F-1hr, W.Q., 1500 F-1hr, W.Q., 900 F-3hr, A.C.	250	260	270	357	13	45	17	52
REP* 300 Grade Extruded at 400 F, 100 in./min and Water Quenched	900 F-3hr, A.C.	265	272	277	386	12	46	13	52
	1500 F-1hr, 900 F-3hr, A.C.	275	282	284	378	10	46	7	53
	1700 F-1hr, W.Q., 1500 F-1hr, W.Q., 900 F-3hr, A.C.	254	264	272	373	13	50	10	52
REP* 300 Grade Extruded at 1400 F 400 in./min and Water Quenched	900 F-3hr, A.C.	266	274	276	367	12	49	13	52
	1500 F-1hr, 900 F-3hr, A.C.	272	279	282	365	11	44	9	53
	1700 F-1hr, W.Q., 1500 F-1hr, W.Q., 900 F-3hr, A.C.	254	262	272	359	14	50	10	51

*Rotating Electrode Processed Powder

W.Q. - Water quenched

A.C. - Air cooled

*It is felt that 1450 F - 400 in./min falls midway between 1450 F and 1600 F due to the additional adiabatic heating at the faster rate of extrusion.

CONCLUSIONS

1. Use of rapidly cooled 18Ni Mar 300 powders alleviates alloy segregation banding.
2. Extrusion temperatures must be in the recrystallization range to control fibering of TiN and TiC particles.
3. In this alloy, without "banding" or fibering it is possible to raise the Charpy V-notch toughness from 7 to 23 ft-lb at the same or higher strength levels.

ACKNOWLEDGMENT

The author wishes to acknowledge the able assistance of Mr. J. D. Colgate in the preparation of samples for testing and transmission electron microscopy and to Mr. F. J. Rizzitano for his many suggestions.

LITERATURE CITED

1. DECKER, R. F., EASH, J. T., and GOLDMAN, A. J. *18% Nickel Maraging Steel*. ASM Trans. Quart., v. 55, 1962, p. 58.
2. CHILTON, J. M., and BARTON, C. J. *Identification of Strengthening Precipitates in 18Ni (250) Aluminum, Vanadium, and Titanium Maraging Steels*. ASM Trans. Quart., v. 60, 1967, p. 528.
3. MARCUS, H., SWARTY, J. H., and FINE, M. E. *A Study of Precipitation in Stainless and Maraging Steels Using the Mossbauer Effect*. ASM Trans. Quart., v. 59, 1966, p. 468.
4. REISDORF, B. G., and BAKER, A. J. *The Kinetics and Mechanisms of the Strengthening of Maraging Steels*. Technical Report AFML-TR-64-390, 1965, Wright-Patterson AFB, Ohio.
5. FLOREEN, S., and SPEICH, G. R. *Some Observations on the Strength and Toughness of Maraging Steel*. ASM Trans. Quart., v. 57, 1964, p. 714.
6. PETERS, D. T., and CUPP, C. R. *The Kinetics of Aging Reactions of 18% Ni Maraging Steels*. Trans. AIME, v. 236, 1966, p. 1420-1429.
7. MILLER, G. P., and MITCHELL, W. I. *Structure and Hardening Mechanisms of 18% Nickel-Cobalt-Molybdenum Maraging Steels*. J. Iron and Steel Inst., v. 203, 1965, p. 899.
8. TUFFNELL, G. W., and CAIRNS, R. L. *18% Nickel 350 Maraging Steel*. ASM Trans. Quart., v. 61, 1968, p. 798.
9. SPAEDER, G. J., BROWN, R. M., and MURPHY, W. J. *The Effect of Hot-Rolling Variables on the Fracture Toughness of 18Ni Maraging Steel*. ASM Trans. Quart., v. 60, 1967, p. 418.