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THE EFFECT OF Ti CONTENTS ON THE AMOUNTS OF INCLUSIONS FORMATION AND MECHANICAL PROPERTIES OF C300 HIGH STRENGTH STEEL

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Abstract: *In this research the influence of Ti contents on the amounts of inclusions formation and mechanical properties of a high alloy high strength steel, C300, has been investigated. For this purpose several bars were casted under the same solidification conditions, but different amounts of Ti element. All the seven casted bars were homogenized at 1200 °C for a period of 2 hours. Then, they were immediately hot rolled after homogenization; so that the out rolling temperature was kept in the range of 1000-1200 °C. The specimens were then solution annealed at 820 °C for 1 hour and finally they were aged for a period of 3 hours at 500 °C. The samples were subjected to tensile, impact and hardness tests in order to relate the variation in volume percent of inclusions due to different amount of Ti, to mechanical properties. The results showed that by increasing the amount of Ti a serious decline in toughness properties of the alloy due to increase in inclusion population occurred. So this research provides a very useful information about the relation between volume fraction of inclusions and mechanical properties of a C300 high strength steel.*

Key words: *high strength steel, inclusion, Ti contents, casting, mechanical properties, Maraging C300*

1. INTRODUCTION

High alloy high strength maraging steels having a large amounts of alloying elements such as Ni, Co and Mo belong to high strength martensitic steel group. The hardness of these types of steels before subjecting them to optimization process in comparison to those of other high alloy steels are low, due to their low carbon content, however their hardness and strength could be improved by subjecting these steels to age hardening operation. These types of steels are divided into two groups, one having Co in their compositions and the other is free from Co. The Co group has been classified into four different types, i.e. C200, C250, C300 and C350. The one without Co has been classified into T200, T250 and T300 types [1-3]. These types of steels are generally produced by one of the two following routes [4]:

a) Melting the steel constituents in air and then remelting the products by arc under vacuum.
b) Induction melting the steel constituents under vacuum following by vacuum arc melting.

The objective of melt refining is reduction of unwanted elements such as C, O, N and S as far as possible, hence reduction of inclusions size and their amounts [5]. It has been reported [6] that the toughness of high strength maraging steels seriously can be affected if high density of impure elements is present in the steel compositions. In addition, impurities make the production of high Ti contents high strength steel very difficult. Therefore in order to reduce impurities one can employ various techniques such as vacuum induction melting (VIM), vacuum arc remelting (VAR), electro-slag remelting (ESR) and electron beam melting (EBM). It has been reported [6] that reduction of impurity elements due to use of new processes is much more than that of conventional casting. Typical reduction of unwanted elements has been reported so far [6] are shown in Table 1:

Table 1. Typical reduction of unwanted elements.

Elements	Typical Element Density After Reduction by Convention Technique (ppm)	Typical Element Density After Reduction by New Processes (ppm)
C	300	20
S	130	30
N	100	20
O	100	20

Typical largest inclusion size found in various processes is reported below [6].

Melting Process	The Largest Size of Observed Inclusion (µm)
VIM/VAR	165
VIM/ESR	102
VIM/EBM	23

Formability of high strength steel bars can be increased by reducing alloying elements segregation via homogenization processes [1, 3]. Hot forming improves mechanical properties of these steels due to refining primary austenite grain, preventing formation of soft zones and large precipitates in primary austenite grain boundaries, homogenizing the distribution of inclusions and reducing their sizes, and finally producing fine martensite structure [1, 3].

These types of steels are normally subjected to austenizaing process at high temperature after hot forming and then air cooled, so that fine structure martensite is produce [1, 8]. After solution annealing process, age hardening process in the range of 455- 510 °C for a period of 3-12 hours is usually applied on these steel to obtain very high strength and good toughness properties [1, 3].

The amount of impurities in maraging steels is very low compare with other high strength steels. Low impurities are a recommended requirement in high strength steels for getting a suitable toughness. Among the most harmful elements in high strength steels are S, O, N and C [1, 3, 6]. These elements cause a decline in mechanical properties such as strength and toughness by forming carbides, nitrides, sulphides and oxides [1, 9]. According to pervious researches [10, 11] inclusions in steels are classified in three groups.

a) Large inclusions having sharp corners, such as Ti (C, N). These inclusions can also have other elements, such as Mo and Zr. If the amounts of C and N in steel do not exceed 0.002 and 0.005 percent respectively, then volume fraction of these types of inclusions will be about or less than 0.001. It has been reported [10], that the volume fraction and sizes of these inclusions can be reduced even more, by remelting the steel bars under vacuum processes.
b) Small inclusions such as TiC and (Ti, Mo)C having mostly cubical shape. These types of inclusions are seen in high strength steels, having high content of Ti and C, [10, 12].
c) Sulphide inclusions, such as TiS, are not seen in all maraging steels. Brirklo et al [10] have reported the sulphide inclusion in high strength steels are of TiS types, while some other researches mostly stated that these inclusions are of Ti2S and Ti2CS types. In addition, it has been said that Ti2S may in fact be Ti_2CS which may erroneously reported to be Ti_2S.

Birkle et al [10] also reported that plate shape precipitates of TiC and inclusions such as TiN, AlN and ZrN may be observed in grain boundaries of some high strength steels. Typical shapes of Ti2S and Ti (C, N) inclusions are presented below [10].

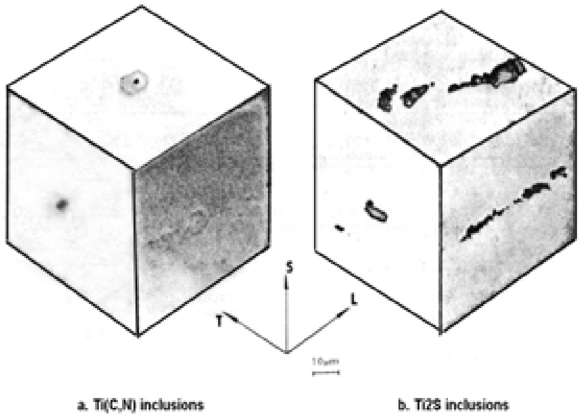

Fig. 1. Ti_2S and Ti (C, N) inclusions shapes in high Ti high strength steel [10].

There are two general methods by which one can either eliminates or modifies inclusions in maraging steels.

a) Eliminating inclusions before producing bars
b) Modifying inclusions after producing bars

For inclusion elimination before production of bars, one should control the purity of charging materials, as well as casting atmosphere, in order to minimize the harmful elements, hence elimination of inclusions [1]. One should notice that for preventing the entrance of harmful elements to casting atmosphere the melting and casting processes of maraging steels should be done under Argon or vacuum [3].

For modification of inclusions after production of bars, thermo-mechanical processes are used as it was mentioned before [1, 3, 15].

Table 2. Chemical analysis of the casted bars, weight percent.

	C	S	Al	Ti	Mo	Co	Ni	Fe
No. 1	0.01	0.012	0.18	0.68	4.51	9.4	18.3	Rem.
No. 2	0.013	0.013	0.125	0.711	4.6	9.35	18.36	Rem.
No. 3	0.012	0.011	0.08	0.799	4.53	9.2	18.14	Rem.
No. 4	0.013	0.012	0.1	1.036	4.31	9.33	18.31	Rem.
No. 5	0.011	0.012	0.11	1.065	4.4	9.35	18.27	Rem.
No. 6	0.014	0.012	0.134	1.48	4.41	9.39	18.36	Rem.
No. 7	0.011	0.01	0.16	1.963	4.38	9.41	18.15	Rem.
Standard	0.03	0.01	0.05-0.15	0.5-0.8	4.6-5.2	8-9.5	18-19	Rem.

2. EXPERIMENTAL PROCEDURE

For investigating the effects of Ti on the amounts of inclusions formation and mechanical properties of maraging steel C300, the constituents materials of this type of steel was melted in a special order in vacuum induction furnace. During melting process, the compositions of the alloy was checked several time and acted upon observed results to correct the melt compositions. Temperature of the melt was controlled by pyrometer. The melt temperature before casting the bars was in the range of 1550-1580 °C. The mould was made of carbon steel St37, having dimensions 300x80x40 mm.

The mould was preheated to 300 °C before the molten alloy was casted into it, and then cooled in air. Table 2 shows the nominal chemical analysis of each casted bar which was measured by quantometer.

Several samples were prepared from casted alloys for rolling. These samples were solution treated at 1200 $\pm$10 °C for a period of 2 hours and then they were subjected to hot rolling process for 60% reduction in thickness via three passes. The samples were quenched after hot rolling in order to obtain martensitic structure and super saturation of alloying elements, in the matrix. Then the rolled and quenched bars were subsequently annealed at 820 $\pm$5 °C for one hour in air. Several subsize tensile specimens according to standard ASTM E8 and impact specimen according to standard ASTM A370 were prepared for the annealed materials. Finally the tensile and impact samples were age hardened at 300$\pm$ 2 °C for 3 hours in furnace before performing the mechanical testing.

3. RESULTS AND DISCUSSION

Structure study of variously bars by light and scanning election microscopes showed that the morphology of inclusions in all casted bars were generally polygonal and occasionally they appear in spherical and elliptical forms. Typical inclusions shapes and distribution in the casted bars are presented in Fig. 2. Table 3 and Fig. 3 show that while the number of inclusions increased with increasing the Ti content of the alloy, the sizes of inclusions were not affected greatly by addition of more Ti. The inclusions sizes in all casting bars were in the range of 3.8 to 8 μm. One can observe in Table 3 that by increasing the Ti content from 0.68 wt % to 1.99 wt % the number of inclusions in one square millimeter increaseed from 200 to 366. Typical shape, size and distribution of inclusions in hot rolled bars are presented in Fig. 4 and the change in the average volume percent of inclusions with Ti content of different cast after hot rolling is shown in Table 4.

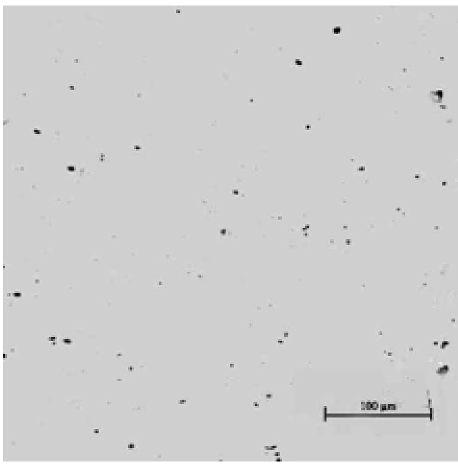

Fig. 2. Typical inclusions shapes, sizes and distribution in cast alloys.

Table 3. The result of quantitative metallography of inclusions and its relation with Ti content in various cast.

Melt No.	Average Ti Content (Wt%)	Average Volume Percent of Inclusions	Average Number of Inclusions Per
1	0.68	0.65	200
2	0.711	0.66	209
3	0.799	0.68	214
4	1.036	0.94	247
5	1.065	0.96	258
6	1.408	1.51	311
7	1.962	1.82	

Typical shape, size and distribution of inclusions in hot rolled bars are presented in Fig. 4 and the change in the average volume percent of inclusions with Ti content of different cast after hot rolling is shown in Table 4. Inclusions sizes in rolled bars vary between 1.1 and 1.6 μm. Comparison of the inclusion sizes in cast and rolled bars indicate that the sizes of inclusions reduced substantially, i.e. about 40%, after rolling the bars. As it was stated before, the most important inclusions in maraging alloy are TiC, Ti_2S and Ti(CN). The shape of TiC inclusions is generally spherical, Ti2S inclusions are usually spindle or fusiform and Ti (C, N) are polygonal and occasionally they appear in spherical morphology. Fig. 5 shows typical inclusions shapes in cast 7 after rolling. Typical EDX analyses of these inclusions are presented in Fig. 6. Considering these results and those presented in some other references [10, 12], it seems, that the inclusions observed in this research are of TiC, Ti2S and TiN types.

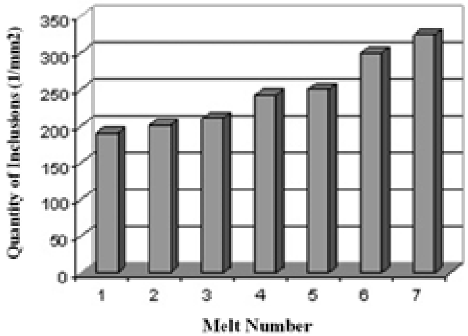

Fig. 3. Bar chart of inclusion number in various cast having different amount of Ti.

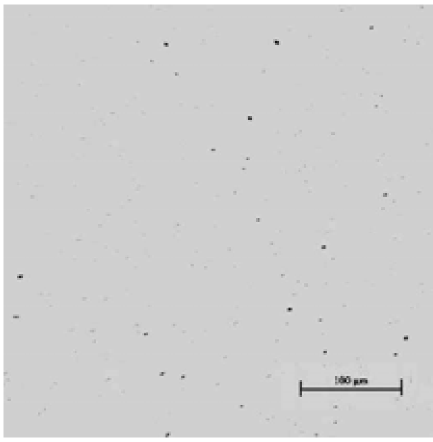

Fig. 4. Typical size, shape and distribution of inclusions in hot rolled bars.

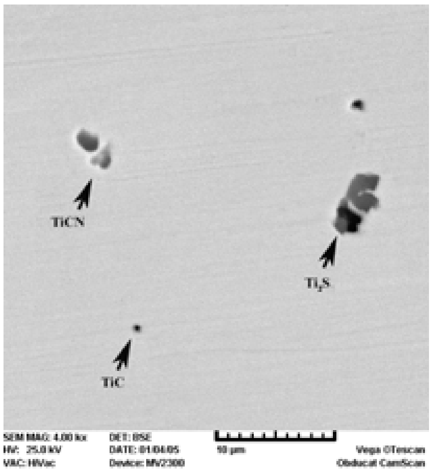

Fig. 5. Various types of inclusions in cast 7.

Worth mentioning that point analysis by SEM used for analyzing inclusion, in this research was not a very reliable method for getting the exact stoichiometric compositions of these inclusions, however, one can use this method to interpret the spectrograph shape changes in point compositions as inclusions compositions richness in some indicated elements.

Table 4. Variation in rolled bars inclusions amounts having different Ti content.

Melt No.	Average Ti Content (Wt %)	Average Volume Percent of Inclusion %
1	0.68	0.62
2	0.711	0.63
3	0.799	0.65
4	1.036	0.89
5	1.065	0.91
6	1.408	1.22
7	1.962	1.41

Table 5. The results of EDX analysis of various types of inclusions observed in cast 6.wt%.

	Fe	Ti	C	S	N	Co	Ni	Ca	Al	O	Mo
Ti_2S	45.88	25.13	-	11.25	-	5.58	9.28	0.15	0.27	2.4	-
TiC	37.11	26.12	22.29	-	-	6.01	8.11	0.35	-	-	-
TiCN	26.59	29.69	15.1	-	13.76	4.47	7.07	-	-	-	3.39

Therefore, comparing the composition of matrix and that of both matrix and inclusions, one may get the inclusions compositions relatively.

Now by considering the kind of elements exist in the observed inclusion, and their deforming behavior in the rolling process and also considering the results of similar researches [10, 12], one may conclude that the inclusions observed belongs to one of the following four groups.

Ti_2S inclusions having fusiform shape. The size of these inclusions is between 6 to 8 µm.

1. TiC's and TiCN inclusions, having either polygonal or spherical shapes. The approximate size is in the range of 3- 5 µm.
2. TiC inclusions which are very small, i.e. 1-2 µm, in comparison to the other inclusions, and having spherical shape.

Al_2O_3, TiN, AlN and (Ti, Mo)C inclusions. These types of inclusions were not observed in this research, but their observation on similar researches have been reported.

Comparing the average sizes of inclusions in the cast and rolled bars one can see a large decrease, about 40%, in inclusions sizes after rolling the cast bars has occurred. In addition the number of inclusions in rolled bars was substantially decreased. This is said to be due to solution of very fine particles and partial solution of large particles during hot rolling process and subsequent annealing operation at high temperature. Also it seems that some modification of the shapes of non-spherical inclusions has occurred during homogenization operation, so that large fusiform inclusions were broken and formed several smaller polygonal inclusions. This observation has also been justified by W. M. Garrison [10] who indicated that the large inclusions usually break during rolling. Quantitative study of inclusions sizes and their volume percent in as cast bars show that by increasing the amount of Ti in melt the number of inclusions and their

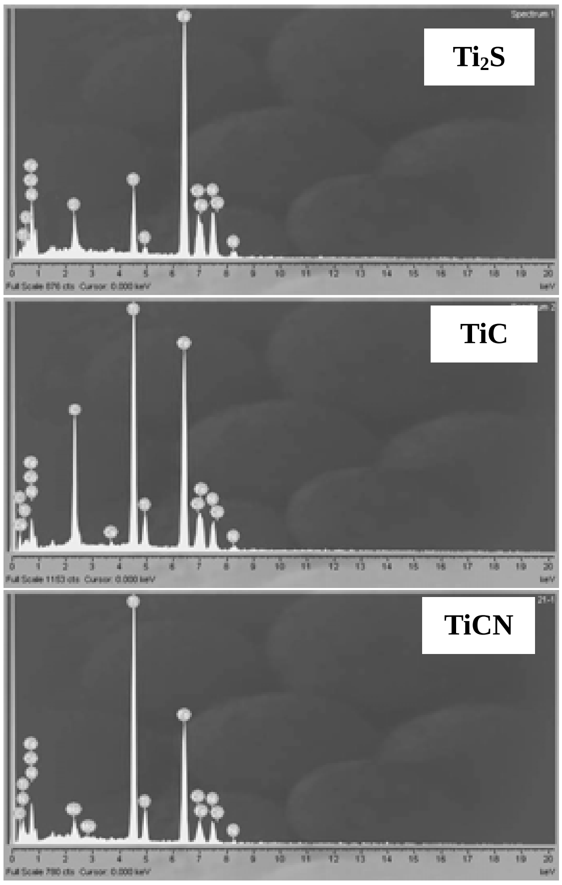

Fig. 6. Spectrographs showing typical analysis of inclusions shown in Fig. 6.

size increase. This could be due to the fact that by increasing Ti content of the melt the possibility of reaction between Ti and impurity elements such as C, N, and S increases and as the result, the chance of forming inclusions such as TiN, Ti_2S and TiN become more. The results of impact charpy test at room temperature are shown in Table 6. These results show increasing the Ti content of the melt caused a decrease in energy absorbed by the cast samples. Fig. 7 shows the toughness of various age hardened specimens as a function of Ti content of the bars. In addition, on the standard toughness of maraging C300 is presented in this figure for the purpose of comparison. This figure shows bars 1 and 2 which have 0.68 wt.% and 0.71 wt.% Ti respectively absorbed more energy than the other bars and the amounts of their energy absorption are comparable to that of standard sample. Therefore by increasing Ti one can expect the toughness and strength of this type of steel to reduce. This could be attributed to the formation of brittle inclusions and precipitates such as Ni_3Ti which their formation not only directly reduces the toughness but also since their formation require a large amount of Ni and the source of providing Ni, is the matrix, thus the matrix become depleted from Ni which by itself is another reason for reduction of the toughness. [15, 16, 17].

Table 6. Charpy impact energy of the bars as a function of Ti content.

Impact Energy (J)	17	15.9	14	11	10.5	6	4
Ti Content (Wt%)	0.68	0.71	0.79	1.036	1.065	1.48	1.96

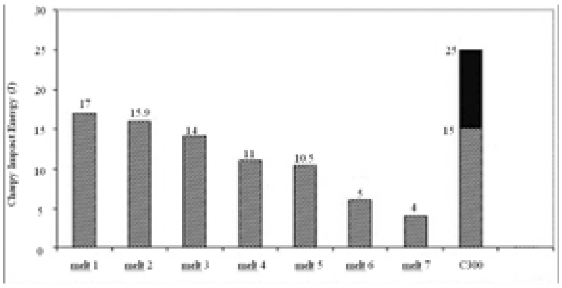

Fig. 7. Impact energy of aged specimens together with the impact energy of standard maraging C300 for comparison.

Figs. 8 and 9 show typical fracture surfaces of casts 1 to 5 which were age hardened. The fracture surfaces of these casts were generally ductile and show characteristic ductile dimples. Cast 7 with maximum amount of Ti, broken in brittle mode, is shown in Fig. 10. This figure shows the chevron pattern with cabage leaves which are good indication of brittle fracture. As it can be seen in Fig. 9 a large number of dimples were associated with particles. Due to the large difference of elastic modules of these particles and the matrix, their interface with matrix become separated when the specimens are subjected to both tensile and compressive stresses [18].

This leads to the formation of plastic hole. These holes enlarged by further deformation lead to ductile transgranuler fracture as it can be seen in Figs. 8 and 9. Typical point analysis of an inclusion shown in ductile fracture surface, Fig. 9, of the specimens is presented in Table 7.

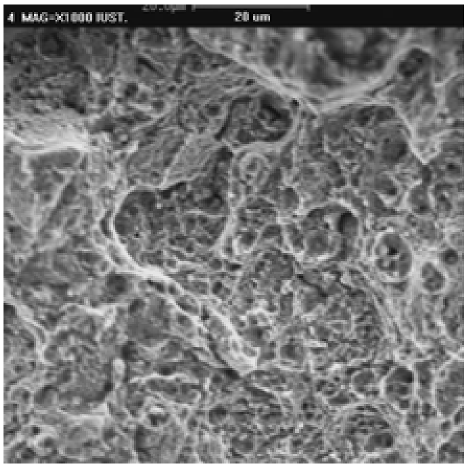

Fig. 8. Typical fracture surface of casts 1 to 5.

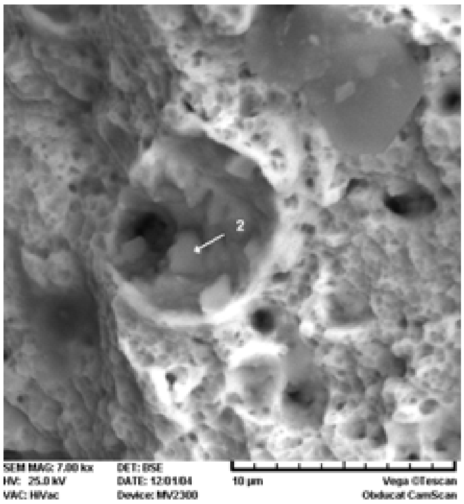

Fig. 9. Typical ductile dimples in the surfaces of fracture surface of casts 1 to 5.

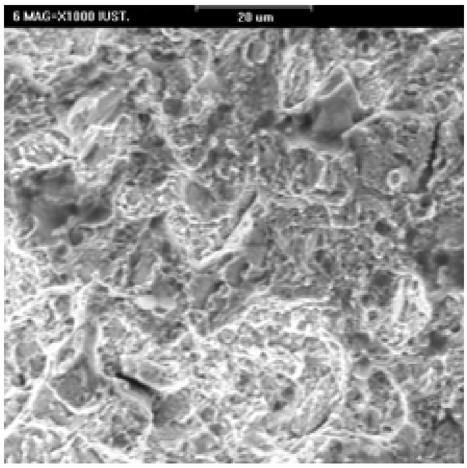

Fig. 10. Typical brittle fracture surfaces of casts 7.

Table 7. Point analysis of inclusion shown in Fig. 11.

Composition	Fe	Ti	C	S	Ni	Co
Wt%	38.16	28.12	13.93	9.54	6.14	4.02

4. CONCLUSIONS

1. By increasing the amount of Ti from about 0.7 wt% to 2 wt% in the melt, the number and volume percent of inclusions increased substantially.
2. Addition of Ti to maraging C300 steel melt caused the formation of fine structure of martensite after rolling process.
3. Ti addition to this type of steel increased their hardness.
4. Strength and toughness of this kind of steel increased with addition of certain amount of Ti up to 0.7 wt.%, however when the Ti content become more than 0.7 wt.% the toughness of steel gradually reduced.
5. The mode of fracture changes from ductile to brittle, when Ti content increases more than 1.5 wt.%.

REFERENCES

1. R. F. Decker, “Source Book on Maraging Steels”, ASM International, Metals Park, OH., 1978, PP.1-19,351-357.
2. ASM Metal HandBook, 10 th ed., Vol.1, ASM International, Metals Park, OH., 1991, PP. 793-800.
3. ASM Metals HandBook,10 th ed., Vol.4, ASM International, Metals Park, OH., 1991, PP.219-228.
4. Dr .S. Floreen and A. M. Bayer,” Development and Commercialization of Cobalt-Free Maraging Steels”, Material Society, 1988 ,pp.39-54.
5. F. B. Pickering, “The Constitution of Non-Metallic Inclusions in Steel”, Published by the British Steel Corporation, Sept. 1974,pp.141-154.
6. R F. Decker and S. Floreen, “Maraging Steels-the First 30 Years” ,Proc.of the Conf. On Maraging Steels Recent Developments and Applications, TMS/AIME, Warrendale, PA,USA,1988,pp.1-30.
7. S. Floreen and A. M. Bayer,” Development and Commercialization of Cobalt-Free Maraging Steels”, Material Society, 1988,PP.441-458.
8. R. F. Decker and S. Floreen, “Maraging Steels-the First 30 Years” ,Proc.of the Conf. On Maraging Steels; Recent Developments and Applications, TMS/AIME, Warrendale, PA,USA,1988,PP.1-30.
9. G. A. Roberts and R. A. Cary, “Tool Steel, 4th ed., American society for Metals”, 1980,pp.194-196.
10. W. M. Garrison, Jr., “Maraging Steels: Structure Toughness Relationship”, Proc. Of the Conf. On Maraging Steels; Recent Development and Applications”, TMS/AIME, Warrandle, PA, USA, 1988, PP.177-211
11. J. A. Psioda, “The Effect of Microstructure And Strength on the Fracture Toughness of an 18Ni,300 Grade Maraging Steel”, PhD. Thesis, Carnegio-Mellon University, 1977,171-187.
12. Technical Note, “Non-Metallic Inclusions in Maraging Steels”, “Journal of The Iron and Steel Institute”, 1972, pp.793-795.
13. Hans Martin Kuhn, “ Remscheid Arch Eisenhuttenues”, Aug.1975, pp.122-131.
14. H. J. Rack and David Kalish, “Improved Fatigue Resistance of 18Ni(350) Maraging Steel Through Thermomechanical Treatments”, “Metallurgical Transactions”, Vol.5, Mar. 1974,pp.57-64.
15. D. M. Vanderwalker, “The Study Of Precipitation In Maraging Steels”, “Proc. Of the Conf. On Maraging Steels; Recent Development and Applications”, TMS\AIME, Warrndale, PA, USA, 1988,PP.255-267.
16. K. Vasudevan, S. J. Kim, and C. M. Wayman, “Precipitation Reactions And Strengthening Behavior In 18Wt pct Nickel Maraging Steel”, “Metall. Trans. A.”, Vol. 21, 1990, PP.2665-2668.
17. U.K.Viswanathan, G. K. Dey and M.K.Asundi, “Precipitation Hardening in 350 Grade Maraging Steel”, “Metal. Trans.”, Vol.24A, 1992, PP.2429-2442.
18. M. F. Ashby, .Gandhiand D. M. Taplin, “Acta Metallurgical ”,Vol.27,1979,pp.699-729.