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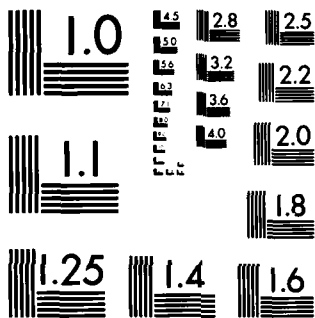
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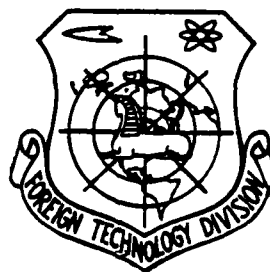
FOREIGN TECHNOLOGY DIVISION



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OF MARAGING STEELS

by

Han Yaowen and Pan Yucheng



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A STUDY ON VACUUM MELTING TO INCREASE THE TOUGHNESS OF MARAGING STEELS

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Abstract

The toughness of maraging steels decreases with the increase of their strength.

This paper focuses research on the factors influencing the toughness and ductility decreases of maraging steels as well as the effects of vacuum induction melting, vacuum arc remelting and electron beam refining techniques on eliminating impurities in maraging steels.

Research results show that the interstitial elements such as C, S, N, O and other impurity elements have great influences on the toughness and ductility of maraging steels. Increase of the Ti(C,N) content in maraging steels can lower their fracture toughness and their reduction of area.

Vacuum induction melting together with the vacuum arc remelting cannot only accurately control the chemical composition in maraging steels but it can also effectively eliminate the harmful impurities in maraging steels. The electron beam remelting technique can more effectively eliminate antimony, lead, tin and other impurity elements in maraging steels and it can also further reduce the carbon and oxygen contents in maraging steels. Therefore, it increases the fracture toughness and ductility of maraging steels.

I. Introduction

Since 1959, a series of maraging steels of 20Ni, 25Ni,

12Ni, 18Ni etc. intensity levels with yielding strength of $140-245\text{kg}\cdot\text{f}/\text{mm}^2$ have appeared. Since the beginning of the 1970's, maraging steels greater than $250\text{kg}\cdot\text{f}/\text{mm}^2$ and even $280-350\text{kg}\cdot\text{f}/\text{mm}^2$ have been researched. Present maraging steels are not of one or two steel types but a series of steel types with yielding strengths of $140-350\text{kg}\cdot\text{f}/\text{mm}^2$ have already been formed. Maraging steel is the highest strength steel type among steels yet its toughness is higher than other ultra-high strength steels with the same intensity level [1]. Maraging steels are also the same as other ultra-high strength steels in that their toughness continuously decrease with the continuous increases of the strength. Therefore, the increase of the toughness and ductility of maraging steels has been an important foreign and domestic research topic in recent years.

There are many factors influencing the toughness of maraging steels [2,3]. Firstly, the alloy elements and structural compositions in the steel are influential. The thermal processing systems and methods of steel as well as pressure machining techniques etc. all show different levels of influence, especially the influence of melting techniques.

We studied four types of maraging steel with $175-280\text{kg}\cdot\text{f}/\text{mm}^2$ intensity levels and a maraging steel series is being made domestically based on intensity level. When studying maraging steel, especially steel of $200\text{kg}\cdot\text{f}/\text{mm}^2$, we encountered the same problems as in foreign research, that is, the raising of the toughness and ductility of steel has been an important research topic in recent years.

II. Test Method and Research Content

The several types of researched maraging steel all have the problem of increasing toughness. This paper focuses research on two types of steel: 175 and $245\text{kg}\cdot\text{f}/\text{mm}^2$. All of the test steel

underwent vacuum induction melting and the tests were carried out in a laboratory and steel plant. For comparative research, we especially selected a furnace model with 0.027% carbon content.

The first type of test steel underwent 1220°C diffusion annealing and was forged into hot-rolled slab. It was hot-rolled at 1080°C heating and rolled into two hot-rolled steel plates of 5.5mm and 2.5mm. The cold-rolled system rolls two cold-rolled steel plates of 3.7 and 1.5mm on a 1200mm flat shape rolling mill.

The test steel went through inclusion grade appraisal and tensile, impact and fracture toughness detection tests were also carried out. Table 1 gives the results of the test steel's chemical composition and inclusion tests.

(1) 试验号	(2) 化 学 成 分, %										(3) 钢中气体, %				(4) 夹杂物级别			(7) 冶炼方法
	Ni	Co	Mo	Al	Ti	C	Mn	Si	P	S	[O]	[N]	[H] ppm	氧化物 (5)	硫化物 (6)	TiN		
318-204	18.25	8.02	5.04	0.1	0.46	0.01	<0.05	<0.05	0.007	0.008	0.002	0.002	<0.1	0.5	0.5	1	真空感应加真空电弧重熔(8)	
318-205	17.99	8.01	5.1	0.1	0.53	0.01	<0.05	<0.05	0.007	0.006	0.001	0.001	<0.1	0.5	0	0.5	真空感应加真空电弧重熔(9)	
318-212	17.33	7.81	4.96	0.045	0.45	0.01	<0.05	<0.05	0.007	0.007	0.0015	0.0012	<0.1	0.5	0	0.5	真空感应加真空电弧二次重熔(10)	
318-213	17.97	7.98	4.73	0.1	0.51	0.01	<0.05	<0.05	0.007	0.007	0.0016	0.003	<0.1	0.5	0	0.5	真空感应加真空电弧二次重熔(11)	
318-214	17.97	8.11	5.03	0.14	0.55	0.027	<0.05	<0.05	0.005	0.008	0.0025	0.001	<0.1	0.5	0	0.5	真空感应加真空电弧一次重熔(12)	
418-454	18.23	7.56	5.0	0.13	0.52	0.015	0.034	<0.05	0.005	0.005	0.002	0.005	1.1	0.5	0	0.5	真空感应加真空电弧一次重熔(13)	

Table 1 The test steel's chemical composition and inclusion grades and the gas contents.
(continued next page)

Table 1 (continued)

Key: (1) Test number; (2) Chemical composition; (3) Gas in steel; (4) Inclusion's grade; (5) Oxide; (6) Sulfide; (7) Melting method; (8)-(9) Vacuum induction with vacuum arc remelting; (10)-(11) Vacuum induction with two vacuum induction arc remeltings; (12)-(13) Vacuum induction with one vacuum arc remelting.

After the second type of test steel undergoes vacuum induction melting, it is remelted in an electron-beam furnace, extruded into a tube blank, spun into a thin walled tube and then we carried out property determination.

III. Test Results and Discussion

We studied the influences of microimpurity elements, steel purity and melting methods etc. on the maraging steel's fracture toughness and ductility and the test results are listed below.

1. The Influences of Microimpurity Elements and Steel Purity on the Maraging Steel's Toughness and Ductility

(1) The Influence of the Carbon Content in Steel

By studying two types of steel with different carbon contents and the K_{1C} value changes with different aging temperatures, we can see that the influences of the carbon content on the maraging steel's fracture toughness is very substantial (Fig. 1). In the figure, (curve 1) is steel with carbon content of 0.01% and the K_{1C} values with various aging temperatures are higher than steel with carbon content of 0.027% (curve 2).

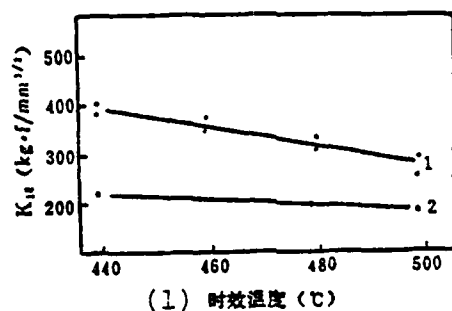


Fig. 1 The influences of different carbon contents on maraging steel K_{1C} : 1 - 0.01%C; 2 - 0.027%C.

Key: (1) Aging temperature.

Electron microscopic observations of the fractures of these two types of steel show that steel specimens with carbon contents of 0.01% have a smaller brittleness phase separated out than that of steel with 0.027% carbon contents. Table 2 shows the chemical analysis results after separation phase extraction.

试样号 (1)	C含量, % (2)	(3) 析出相含量, %		
		TiC	TiN	[N]
318-213	0.010	0.045	0.0052	0.0012
318-214	0.027	0.110	0.0110	0.0026

Table 2 Separation phase analysis results.

Key: (1) Specimen number; (2) Contents; (3) Separation phase contents.

Table 2 shows that the TiC and TiN phase contents in 318-214 steel with high carbon content has multiple increases over 318-213 steel.

The influences of the carbon content in the statistical test steel on the K_{1C} value can further prove the test results shown in Fig. 1 and Table 2. Figure 2 shows the statistical results. We can see from the figure the general trend of the influence of

carbon content on K_{1c} .

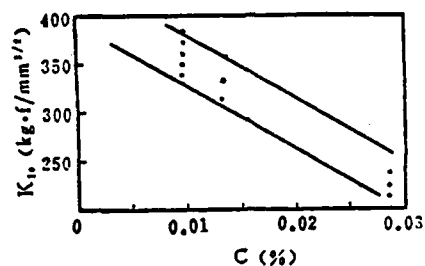


Fig. 2 The influence of the carbon content in steel on its K_{1c} .

In regards to the influences of carbon content on the toughness and ductility of maraging steels, the research results of many authors are basically the same. It is commonly considered that the main brittleness phase in maraging steel is $Ti(C,N)$ [4,5] which is separated out on the austenite crystal boundary during the cooling process. It not only lowers the K_{1c} , K_{1sc} , a_K and γ etc. of the steel but is often the cracking source of the stress structural piece.

(2) The Influence of Titanium on Strength and Toughness

Test results show that when the TiC and TiN contents in steel are high, the fracturing toughness of the steel noticeably decreases as shown in Table 3.

冶炼方法 (1)	(4) 析出物含量, %			C, %	S, %	K_{1c} kg·f·mm ^{-3/2}
	TiC	TiN	[N]			
(2) 真空感应加 真空电弧重熔	0.045	0.0052	0.0012	0.01	0.007	354
						382
真空感应加 电弧重熔 (3)	0.063	0.0092	0.0021	0.017	0.002	215, 245
						218, 214.5

Table 3 The influences of maraging steel's separation phase and impurity content on K_{1c} .
(continued next page)

Table 3 (continued)

Key: (1) Melting method; (2) Vacuum induction with vacuum arc remelting; (3) Vacuum induction with electroslog remelting; (4) Separated out substance content.

The TiC content in the steel has a uniform proportional relationship with the carbon content in the steel. That is, the higher the C content in the steel the higher the TiC content in the steel increases. It can be seen that the influence of the Ti content in the steel on the steel's toughness is the formation of a large quantity of Ti(C,N) after increasing the C and N₂ contents in the steel.

Ti is the strengthening element of this type of steel and we rely on Ti to increase the strength of the steel. That is, the Ni₃Ti separated out through aging strengthens the maraging steels. However, when the C and N₂ impurity elements existing in the steel seize part of the Ti and form Ti(C,N) and cause brittleness of the steel, this decreases the toughness and ductility of the steel.

Research by some people [2] has also proven that following the increase of the Ti content in the steel, its strength sharply rises yet its K_{1C} sharply drops.

ЕХНЕ2П's [6] research considers that the maraging steel's strengthening element Ti content can increase to 1.8%. This can only guarantee its toughness when the impurity elements in the steel are very low. If the steel's purity is low, then the Ti content must not exceed 0.8%.

(3) The Influence of the Sulphur Content in the Steel

In the tests, we used vacuum induction melting and then employed electroslog remelting and the sulphur content in the steel was 0.002%. The steel specimen with carbon content of

0.017% went through vacuum arc remelting and the sulphur content was 0.007%. The steel specimen with carbon content of 0.01% was in proportion to its K_{1C} value. The K_{1C} of the former was far lower than that of the former as shown in Table 3. The TiC and TiN contents in the electroslag remelted steel listed in the table are higher than those of vacuum arc remelted steel. It can be considered that among the two types of test steels with obtained sulphur content ranges, the influence of the Ti(C,N) content on the K_{1C} steel is far greater than the influence of the sulphur content. Therefore, the influence of the sulphur content on steel toughness often requires the uniform consideration of the comprehensive influences of other elements. For example, Cottrell [7] considers that the compound action of S and As in maraging steel can markedly decrease the K_{1C} value of the steel.

The influence of the S content on the toughness of maraging steels should be given serious attention. Based on reports [8], the S content has a relatively large influence on the Charpy impact toughness of maraging steels.

Research by Novak [9] also shows that the S content in maraging steel should be less than 0.01% and he considers that the S contained in the steel exists mainly by the Ti_2S inclusion form, assumes a long strip shape and causes the impact toughness of the steel to have directional decreases.

We added 2.5kg of misch metal per ton of steel in the vacuum induction furnace melting of maraging steels. The desulphurization rate reached 70% and the remaining rare-earth content in the steel fluid gradually decreased with the lengthening of the melting time [10].

(4) The Influence of the Steel's Purity

After undergoing vacuum induction melting with vacuum arc

remelting, the purity of maraging steel is relatively high and the inclusion grades are all below the 0.5 grade and some reach the zero grade. However, the control of the melting technique is unsuitable and there are also exceptions. The K_{1c} value of the above mentioned test steel 318-214 furnace number (Fig. 1, Table 2) is very low. Electron microscopic observations and fracture analysis show that the total content of Ti(C,N) in this steel specimen is prominently higher than 318-213 etc. furnace numbers, its distribution is more concentrated and its dimensions are larger. The obtained electroslag remelted steel has higher C, N_2 , TiC and TiN etc. impurity contents than in vacuum arc remelted steel and therefore the K_{1c} value of the former steel is noticeably low. This was already noticed in Table 3. If we then add O_2 , H_2 , S and some inclusions, the comprehensive influence is even more noticeable.

Some people who have studied the influence of the purity of steel on the toughness of steel [11] used two types of steel with high purity and common purity for comparison tests. The two types of metals contained different impurities and the size and distribution etc. of the inclusions in the steel were also different. Test results show that the steel's purity truly has a large influence on maraging steel. Based on reports [1], the influence of the steel's purity on its impact toughness is also very large.

2. The Influences of the Melting Methods and Techniques on the Toughness of Maraging Steels

Our research results and foreign research reports both consider that the microimpurity elements and inclusions in maraging steels have definite influences on their toughness and ductility. The elimination of microimpurities in the steel and the raising of the steel's purity are determined by the selection of the melting method, improvement of the melting technique

as well as increasing the melting effects. Many authors commonly consider that melting is an innate factor influencing maraging steels and that melting effects determine the toughness of maraging steels [13,14,15,16].

In view of the facts that vacuum induction melting can control the chemical composition of maraging steels and can effectively eliminate harmful impurities in steel, we first studied the carbon-oxygen reaction in the vacuum induction melting process as well as the crucible and molten bath reactions and the alloy element and impurity changing processes etc.

When we use low carbon and low oxygen pure steel containing aluminum as the melted steel raw material, although we do not add a deoxidant and oxidant, we can obtain relatively satisfying carbon and oxygen contents. If we add in Fe_2O_3 decarbonization, this can control the carbon content in the steel to be less than 0.003%. At the same time, the oxygen content can also be about 0.002%. The hydrogen content in the steel is less than 2ppm and the nitrogen content is less than 0.005%. The Mn content in the steel decreases because of volatilization.

In order to further raise the purity of the steel, eliminate the impurities and improve the steel ingot's crystal condition and metallurgical defects, the vacuum induction melted steel then underwent vacuum arc remelting. After investigative research on the electrical parameters and techniques of the vacuum arc remelting process, we can obtain relatively good vacuum arc remelting and purification of maraging steel effects under laboratory and production conditions. Comparative research shows that the K_{1C} of vacuum arc remelted steel is far higher than when using electroslag remelting. The test results are shown in Tables 3, 4 and 5.

(1) 试样号	(2) 冶炼方法	$K_{Ic}, \text{kg} \cdot \text{mm}^{-3/2}$		$\alpha_k, \text{kg} \cdot \text{m}/\text{cm}^2$	
		(4)	(5) 冷轧板材(3.7毫米)	(7)	(8)
		板 坯 (6)	板材试样方向 KQ	板 坯	板 材
318-204	(3) 真空感应加真空电弧重熔	395 386		5.7 7.0	
318-205	"	420, 402 380, 382	纵 (9) 向 444, 458 438, 447	7.0 6.6	8.3, 7.4 9.1
318-212	"	464 524	纵 (10) 向 427, 446 439, 468 横 向 432, 411 (11) 444, 427	7.0 7.5 6.0 7.0	9.0, 8.5 7.8
318-213	"	382 354		6.9 7.0	
318-214	"	224 220.5, 205	纵 (12) 向 407, 383 397, 402 横 向 334, 346 (13) 339, 339	5.7 5.7 5.7	9.2, 9.5 8.0, 8.1 7.7, 7.8
518-454	"	361.0 323.0		6.0 5.7	
518-141	"		320 300	6.3 6.0	

Table 4 Test results of the toughness and impact of $175\text{kg} \cdot \text{f}/\text{mm}^2$ maraging steel slabs and plates.

Key: (1) Specimen number; (2) Melting number;
 (3) Vacuum induction with vacuum arc remelting;
 (4) Slab; (5) Cold-rolled plate (3.7mm);
 (6) Plate specimen's direction; (7) Slab;
 (8) Plate; (9)-(10) Vertical direction;
 (11) Horizontal direction; (12) Vertical direction; (13) Horizontal direction.

We can see that the K_{Ic} value of maraging steel after vacuum induction melting and then vacuum arc remelting is generally above $350\text{kg} \cdot \text{f}/\text{mm}^{-3/2}$ and that the K_{Ic} of steel after vacuum induction with electroslog remelting does not exceed $203-245\text{kg} \cdot \text{f}/\text{mm}^{-3/2}$. The TiN, C and N contents of the former

steel are lower than those of the latter, the inclusion grades are lower and the grains are smaller (Table 2). Aside from the impurity volatilization elimination in the vacuum arc melting process being superior to the electroslag melting process, under arc effects, the inclusions in the steel can be decomposed, refined, float up and be eliminated. These are favorable conditions.

Vacuum arc melted steel often has a banded structure which influences the toughness of the steel, being even more serious for large steel ingots. Use of an electronic probe micro-region to analyze this type of banded structure proves that it is mainly composed from the segregation and enriching of Ni, Mo, Ti etc. elements in the steel. The analysis results are shown in Table 6.

(1) 试 号	(2) 熔炼方法	(7) 化 学 成 分 %						
		C	Si	Mn	Ti	P	[N]	[O]
901 (3)	真空感应加 电渣重熔	0.01	<0.05	<0.05	0.32	<0.005	0.0023	0.007
902 (4)	真空感应加 电渣重熔	0.01	<0.05	<0.05	0.23	<0.005	0.002	0.017
904 (5)	真空感应加 电渣重熔	0.01	<0.05	<0.05	0.64	<0.005	0.0025	0.003
316—291 (6)	真空感应加 电渣重熔	0.017	0.07	0.03	0.51	0.009	<0.005	

(8) 试 号	(9) 熔炼方法	(14) 机 械 性 能				
		σ_b kg·f/mm ²	δ %	ψ %	σ_k kg·m/cm ²	K_{Ic} Kg·f·mm ^{-3/2}
901 (10)	真空感应 加电渣重熔	184 192	10.0 8.0	59.5 64.0	7.3 5.3	304
902 (11)	真空感应 加电渣重熔	172 179	10.5 10.5	62.0 61.0	5.8, 5.6 4.9 5.5	219 203
904 (12)	真空感应 加电渣重熔	203 202	9.0 9.0	58.5 56.5	4.9, 5.6 5.3	243 224
316—291 (13)	真空感应 加电渣重熔	205 210	9.0 9.0	52 51.5	4.9 5.7	215, 214.5 245, 218

Table 5 Mechanical properties of electroslag remelted steel.

Key: (1) Test number; (2) Melting method; (3)-(6)
 Vacuum induction with electroslag remelting;
 (7) Chemical composition; (8) Test number;
 (9) Melting method; (10)-(13) Vacuum
 induction with electroslag remelting;
 (14) Mechanical properties.

(1) 试样部位	(5) 元素含量 %				
	Co	Ni	Mo	Ti	Al
(2) 钢 基 体	9.4	17.3	4.1	0.47	0.04
(3) 白 色 区	9.5	19.1	5.5	0.94	0.05
(4) 带 状 区	9.4	18.2	5.7	0.75	0.05

Table 6 Electronic probe microregion analysis results of banded structure.

Key: (1) Test location; (2) Steel's base body;
 (3) White region; (4) Banded region;
 (5) Element contents.

During the heat machining, this type of banded structure forms an element banded segregation region along the machining deformation direction and assumes marked directionality so as to enlarge the difference of the K_{1C} values of the vertical and horizontal directions. Tests prove that use of homogenized diffusion annealing processing can reduce and even eliminate this type of banded structure. Controlling the melting technique and solidification and crystallization processes are basic measures for eliminating the banded structure. After test findings, it was considered that in the vacuum arc melting process, the current must be controlled to a moderate degree. If the current is too large and the molten bath is excessively deep, this is a major cause of element segregation. If the stable arc coil's magnetic field current is too large, this can also cause element segregation. The magnetic field current of the stable arc coil should use the lowest value so that the stable arc can avoid the appearance of a banded structure. This is in agreement with some foreign research reports [18,19]. As regards vacuum arc remelted maraging steel, the breaking away from the pollution of the refractory and certain

metallurgical defects that guarantees the premise of the steel's toughness has already been confirmed by many authors [20,21,22]. However, maraging steels contain strongly deoxidized elements Ti and Al etc. oxides and generating elements of nitrides all of which remain in the steel in inclusion form. In the refining process, the contents of these inclusions are much higher than their equilibrium values which shows that the refining process sustains dynamic influences. In addition, the impurities and existing form in the steel liquid determine the structure and dynamics of the reaction process. Дюбанов et al [23] used the thermodynamic data of Эллиот [24] and Большов [25] and calculated by eliminating the oxygen content of the C, Ti and Al equilibrium in the steel. The considered that maraging steel melted in a vacuum can only have the carbon-oxygen reaching be carried out smoothly above 1800°C and at $P_{CO} \approx 0.1$ atmospheric pressure and that the thermodynamic stability of the oxide of Ti can then be greatly lower than at 1600°C. However, in the vacuum arc melting process, the molten bath temperature does not exceed 1800°C. The molten bath's surface temperature can be controlled at 1850°C-2000°C in the electron-beam refining process and in a relatively high vacuum the $\{C\} + \{O\} = CO\uparrow$ reaction capability can then forcefully develop.

We cut the vacuum induction melted maraging steel into block steel material, carried out electron bombardment tests and studied the element changes in the steel. Afterwards, we used 150kw electron beams to carry out remelting tests, studied the further development of $\{C\} + \{O\} = CO$, the decarbonization and deoxygenation effects and the removal effects of nonferrous metal impurity elements. On the basis of this, we also carried out remelting tests with a production scale 200kw electron-beam melting furnace. The percentages of the C, O, Pb, As, Sb, Bi etc. elements in the steel decreased before and after 150kw and 200kw electron-beam melting furnace melting are given in Fig. 3.

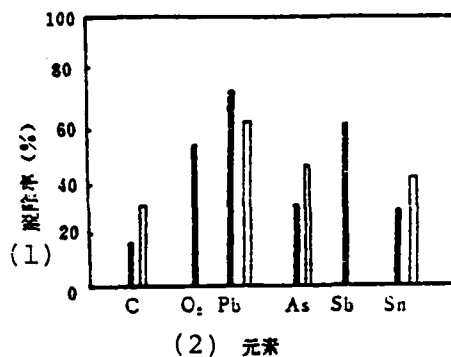


Fig. 3 Removal rate of impurity elements in steel after electron-beam melting (the thick line is the mean value of the removal and the double line is the removal rate of 200kw electron beam furnace melted steel).

Key: (1) Removal rate; (2) Elements.

In the figure, the thick lines indicate the mean values of five test results of the C, Sb, Sn and As elements and the mean values of three test results of the O and Pb elements. The double lines in the figure indicate the test results of the 200kw electron-beam melting furnace. Prior to remelting, each of the contents in the steel were C=0.002-0.017%; O₂=0.0028-0.11%; Pb=0.0003-0.0004%; As=0.0016-0.0034%; Sb=0.003-0.005%; Sn=0.0005-0.0014%. The test results can show that although the C and O₂ contents in the steel are already very low after vacuum induction melting, yet they can noticeably decrease after electron-beam melting. Within this, the C content decreased 5.5-34% and the O₂ content decreased 17.8-70%. The elimination of the Pb in the steel is 66.6-75%; Sb elimination is 50-66.6%; As and Sn have some elimination and only the Bi content has no marked changes. This shows that electron-beam melting can more effectively remove C, O₂ and nonferrous metal impurity elements.

After vacuum induction melting with electron-beam remelting, the purity of the maraging steel is high, the impurity content is low and the toughness is relatively good. When we

compare the cold spun thin wall steel tube made from this type of steel spinning and the same type of steel pipe with an inlet, its quality and major technical indices all reach the inlet steel level. For example, the yield-strength ratio (σ_{Nb}/σ_b) is 1.08 (the inlet steel is 0.65-1.0) and the stress concentration coefficient is 4.0 (the same as the inlet steel). The mechanical properties, hot extrusion, cold spinning and other technical properties of this type of steel all reach the safe level as those reported abroad.

IV. Conclusion

Although maraging steel has better toughness than other ultra-high strength steels with almost the same intensity level, yet its toughness correspondingly decreases with the increase of the strength. It is still an important topic of present research.

Research proves that minute quantities of carbon, oxygen, nitrogen and inclusions in metal all influence the toughness of the metal. The purity of the metal, especially the melting method are crucial to influencing the steel's toughness to decrease. The Ti(C,N) inclusions in the steel is one main reason for the steel's K_{1c} value to drop and brittleness fracturing. The major method for raising the toughness and ductility of the steel is the study of the melting methods and increasing the purification melting effects. Maraging steel using vacuum induction melting with vacuum arc remelting can partially attain purification melting effects yet the purification effects are limited. Maraging steel using vacuum induction melting with electron-beam remelting is now considered to be the most effective method and it is also an effective means for increasing the toughness of steel.

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