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### Anisotropy in Plain Strain Fracture Toughness (K<sub>ic</sub>) Property of Maraging Steels

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#### ABSTRACT

Maraging steels are special class of steels having excellent strength coupled with good fracture toughness. These materials are considered as most suitable candidate aeronautical and missile *materials* for applications for which a good combination of tensile strength and fracture toughness are prime requirements. Maraging steel of grade C 250 has been chosen for present study. The plain strain fracture toughness  $(K_{IC})$  values in longitudinal and tangential orientation of crack propagation display anisotropy for single aged starting material. The extent of anisotropy decreases as the temperature of second aging treatment increases during double aging. The optimum isotropy in fracture toughness is achieved with second aging at a temperature of 485°C. The reason for maximum isotropy in fracture toughness values by re-aging treatments at 485°C / 6 Hrs can be attributed to transformation of higher volume fraction of martensite to precipitates during re-aging, which in turn completely eliminate the impression of flow lines formed

during extrusion (or) prior deformation processes.

*Keywords*:— *Maraging steels, Double aging, Tensile Strength, Fracture toughness (K\_{IC}),* 

#### I. INTRODUCTION

Maraging steels are low carbon Iron-Nickel alloys with additional alloying elements of Cobalt. Molybdenum, Titanium and Aluminium. These are precipitation material used for many strengthened applications in wrought forms due to its combination exceptional of strength. toughness, excellent good formability, machinability and weldability [1]. Another important characteristic of Maraging steel is fracture toughness property which is also an area of interest for rocket motor casing material [2-4]. The optimum values of fracture toughness can be achieved for a particular grade of Maraging steel as a function of heat treatment has already been established [5-14]. Marging steel of grade C 250 (AMS 6512) is used extensively in single aged condition and display excellent

combination of strength, ductility and toughness. But the problem which is frequently encountered by designer is anisotropy in tensile strength and fracture toughness properties. It has been shown that the fracture toughness of Maraging steel varies with specimen orientation i.e. the Maraging steel displays anisotropy in fracture toughness. The anisotropy in fracture toughness misleads the designer to realize a component having requirement of isotropic properties. This generally over estimates (or) underestimates the designer's data. The anisotropy in tensile properties for tube material is generally judged by their 0.2% proof strength and UTS values with respect to longitudinal and tangential orientations.

Several ways have been suggested to arrest cracks in particular direction to make fracture toughness an isotropic property in various alloys, but there are scarcity of literature in the field of anisotropy of fracture toughness of Maraging steel. Therefore, an attempt has been made to suggest a technique to reduce the extent of the anisotropy in fracture toughness while maintaining the same level of typical strength. In this process, the effect of second aging on fracture toughness and tensile behaviour of maraging steel has been studied extensively.

#### **II. EXPERIMENTAL WORK**

The present work entitled "Anisotropy in plain strain fracture toughness  $(K_{IC})$ property of maraging steel of grade C 250" is an attempt to address the above issues. This experiment aims to analyse the tensile flow and work hardening behaviour of flow formed and flow formed plus aged high maraging steels strength through constitutive relations and correlation of microstructures with mechanical properties. An attempt has been made to suggest a technique to make the Maraging steel

isotropic with a suitable heat treatment while maintaining the same level of strength without deteriorating its ductility. for mechanical The specimens and were metallographic tests taken from starting material, flow formed and flow formed plus aged material as per the flow sheet of experiment shown in Figure 1.



#### Figure 1: Flow Chart of Experiment

Material obtained from double vacuum treatment *i.e* VIM(Vacuum Induction Melting) & VAR (Vacuum Arc Remelting) was extruded to a tube of Maraging steel of grade C 250 having of size OD (Outside Diameter) -375 mm, ID (Inside Diameter) -340 mm with 570 mm length. This extruded tube was double homogenised at 950°C / 2 Hrs / Water quenching followed by solution annealing at 820°C / 3 Hrs / Air cooling (AC). Chemical composition of starting material was evaluated using optical emission spectroscope (OES, Make: Spectrolab M10) for all important elements except carbon and sulphur. The carbon and sulphur contents were analysed by using Leco CS600 analyser.

As shown in figure 2 samples were selected from extruded tube for study the effect of double aging on fracture toughness and tensile properties. Initially, the starting material was aged at temperature 485°C / 6 Hrs / Air cooling for evaluation of tensile and fracture toughness properties and designated as numeral 1 throughout the thesis. Subsequently, these aged samples were pre-cracked and re-aged at three different aging cycles. These three different aging cycles were denoted using numerals 2 (150°C / 6 Hrs / Air cooling), 3 (250°C / 6 Hrs / Air cooling) and 4 (485°C / 6 Hrs / Air cooling) along with some prefix indicating the type of test referred in that section of the thesis, respectively.

Fracture toughness sample



Figure 2: Schematic representation of sample extraction plan from extruded tube.

#### **Tensile** Test

Room temperature tensile test was carried out on round specimens selected from extruded tube material. Tensile test was carried out as per standard ASTM E 8 M on round specimen as shown in figure 3. In present study Instron 8500 universal testing machine was used for tensile test. An extensometer of Instron make having gauge length of 25 mm and calibration class of 0.5 was used to measure average linear strain with accuracy of  $1 \times 10^{-4}$ . The crosshead speed maintained during test was 0.5 mm / min so as to maintain the strain rate of 0.0088 mm/mm/min which is within the range specified in ASTM standard.

The starting material in single aged condition was designated as TL1 and TT1, where the second alphabetical letters L and T denotes directionality of the specimens that is longitudinal and tangential, respectively. The suffix 1 represents single aging of starting material. Similarly, the double aged specimens were designated as TL2, TT2; TL3, TT3 and TL4, TT4 corresponding to tensile specimens in 2, 3 and 4 aged conditions, respectively.

Gauge Length(G)25.0 mm Diameter (D) 6.25 mm Radius(R) 5 mm Reduced section length (A)32 mm



Figure 3: Round tensile specimen drawing [ASTM E 8 M]

#### Fracture Toughness Test

Plain strain fracture toughness test was carried out with a CT (compact tension) specimen as shown in the figure 4. The specimens were extracted from starting material and aged with a heat treatment cycle of 485°C / 6 Hrs / Air cooling. Subsequently, all these aged specimens were fatigue pre-cracked as per the cycles given in ASTM standard. The pre-cracked

specimens were then subjected to different aging treatments. The specimens with single aging treatment denoted as FTCL1 and FTLC1 were directly subjected to fracture toughness testing using machine of make Waltar Bai W-B as per the standard ASTM E 399. The letters FT denotes fracture toughness, whereas letters CL and LC are the crack plane orientations defined in ASTM E 399 standard as shown in Fig. 5. Similarly, the designation for double aged specimens was given as FTCL2, FTLC2; FTCL3, FTLC3 and FTCL4, FTLC4 for samples corresponding to second aging cycle of 2, 3 and 4.



Figure 4: Fracture toughness specimen drawing [ASTM E 399]



Figure 5: Schematic of crack plane orientation for hollow cylinder [ASTM E 399]

#### Scanning Electron Microscopy

Fracture toughness tested specimens were characterized by using scanning electron microscope of make Carl Zeiss of model EVO 18 attached with EDX facility. Fracture study was carried out in secondary electron mode. Energy dispersive X-ray spectroscopy was carried out during SEM analysis to find out chemical composition of some particles in the matrix.

#### **III. RESULTS AND DISCUSSIONS**

The analyzed chemical composition of starting material is given in Table.1. The chemical composition of each element is given in weight percent and lie well within the range of specification of Maraging steel grade C 250.

As shown in Figure 6 the microstructure of annealed sample displays homogeneously distributed lath martensite. The average grain size of annealed samples calculated using linear intercept method of ASTM E 112 is ~ 65  $\mu$ m. The starting material in aged condition consists of colony of lath martensite.



Figure 6: Optical micrograph of (a) Solution Annealed and (b) Aged

### TABLE 1: CHEMICAL COMPOSITION OF EXTRUDED AND SOLUTION ANNEALED MARAGING STEEL OF GRADE C 250

Elements Wt %	С	Si	Mn	Ti	S	Р	Ni	Al	Cr	Со	Mo	Fe
Specified val- ues in	0.03 Max	0.1 Max	0.1 Max	0.3 0.5	0.01 Max	0.01 Max	17.0 19.0	0.05 0.15	0.5 Max	7.0 8.5	4.6 5.2	Bal.
Obtained val- ues	0.005	0.005	0.01	0.44	0.002	0.005	18.09	0.11	0.01	8.04	4.89	Bal

#### **Tensile Properties**

## Solution Annealed and Aged extruded tube material

The tensile parameters of extruded tube in solution annealed and aged material are given in Table 2. It can be seen that after aging the strength values are significantly improved while the elongation is reduced. The 0.2% proof stress values are almost same for both longitudinal and tangential aged specimens. The UTS and elongation values moderately decreases and slightly increases, respectively in tangential direction as compared to axial orientation.

#### Single and double aged material

Tensile test parameters of starting materials evaluated on single and double aged specimens in both Longitudinal (L) and Tangential (T) directions are given in Table -3. It is seen that the 0.2 % proof stress values are always lower in T direction as compared to L. The overall proof stress values remain nearly same for single and double aged conditions except for specimens TL4 and TT4. The values of 0.2% proof stress are moderately high for TL4 and TT4 specimens. Similar variation can be observed for values of UTS. However, the values of percent elongation remain almost same for all the specimens subjected to either single or double ageing.

The engineering stress - strain curves for single and double aged specimens in L and T orientations have been shown in Figures. 7 and 8. Both the L and T specimens display continuous and uniform decrease in stress with increase in strain till fracture. It is evident that the double aged specimens TL4 and TT4 have higher values of stresses as compared to other specimens after yield stress. This difference is very clear for TT4 as compared to TL4 specimens as can be seen in the inset figures provided within the figures 7 and 8.

 Table 2: Tensile Test Parameters of Extruded Tube in Solution Annealed and Aged Conditions.

	Tensile test parameters				
Condition of material	0.2% Proof stress in MPa	Ultimate ten- sile stress (UTS) in MPa	% Elongation at gauge length 4*Diameter		
Extruded and Solution annealed (longitudinal direction)	865	1045	19		
Extruded and Solution annealed + aged (longitudinal direction)	1725	1786	13		
Extruded and Solution annealed + aged (tangential direction)	1726	1778	14		

Table 3: Tensile Test Parameters of Single and Double aged Materials in Longitudinal and
<b>Tangential Direction</b>

	<u> </u>	Tensile test parameters				
Condition of material	Sample identifica- tion	0.2% Proof stress in MPa	Ultimate ten- sile stress (UTS) in MPa	% Elongation at gauge length 50 mm		
Solution Annealed and aged at 485 °C / 6 Hrs /AC	TL1	1727	1786	13		
	TT1 1726 1778		14			
Solution Annealed and aged at 485 °C / 6 Hrs /AC	TL2	1744	1805	14		
aging at 150°C X 6 Hrs AC	TT2	1728	1782	14		
Solution Annealed and aged at 485 °C / 6 Hrs /AC + Fatigue cracking + Re-	TL3	1733	1788	13		
aging at 250°C / 6 Hrs / AC	TT3	1726	1782	14		
Solution Annealed and aged at $485 ^{\circ}\text{C} / 6 ^{Hrs} / 4 ^{C}$	TL4	1785	1844	13		
+ Fatigue cracking + Re- aging at 485°C / 6 Hrs / AC	TT4	1780	1841	13		

# Table 4: Voce's Fitted Flow Curve Parameters of Single and Double aged Specimens in L andT orientations.

Condition	Specimen Identifica- tion	s s (MPa)	s <sub>0</sub> (MPa)	-K <sub>V</sub> (MPa)	(R <sup>2</sup> )*
Single agod starting material	TL1	1823	1605	552	0.995
Single aged starting material	TT1	1810	1564	652	0.997
	TL2	1843	1625	570	0.992
	TT2	1817	1620	576	0.999
Deril 1 1. de stin e medenie 1	TL3	1824	1591	639	0.995
Double aged starting material	TT3	1818	1570	601	0.993
	TL4	1871	1661	587	0.999
	TT4	1873	1684	605	0.995



Figure 7: Engineering stress - strain curves in L orientation for single and double aged specimens of starting material.



Figure 8: Engineering stress - strain curves in T orientation for single and double aged specimens of starting material.

The true stress - strain curves for single and double aged materials have been shown in Figure 9. The flow curves in L orientations are overlapping each other in elastic regime in L orientations for both single and double aged specimens. However, in the complete plastic strain regime TL4 is observed to have higher stresses up to UTS as compared to TL1, TL2 and TL3 specimens (Fig. 9a). On the other hand, the elastic and plastic regimes are not overlapped in T orientation for both single and double aged specimens. The flow stress curve for TT4 specimen is observed above the flow stress curves of other specimens such as TT1, TT2 and TT3.

True stress and true plastic strain curves for single and double aged specimens in L and T orientations are given in Fig. 10(a-b). An attempt has been made to best fit the experimental flow curves data. For this, a number of models, such as Hollomon, Ludwik, Ludwigson etc., have been tried but finally it was observed that the Voce's relation fits best with  $R^2$  values between 0.992-0.999 and 0.993-0.999 for L and T specimens, respectively which reflect an adequate fit of flow curves data.



*Figure 9: True stress - strain curves of single and double aged specimens of starting material in (a) L and (b) T orientations.* 



Figure 10: True stress - plastic strain curves in (a) L and (b) T orientations for single and double aged specimens of starting material.

Flow curve parameters of all the single and double aged specimens in L and T direction is fitted with (Eq.1) and corresponding values of flow parameters ( $\sigma_{s}$ ,  $\sigma_{0}$  and  $K_{v}$ ) are given in Table-6. The numerical value of K<sub>v</sub> decreases for L specimens from TL1 to TL3 and then slightly increases for specimen TL4. In contrast, the values of  $K_v$ increases and then decreases from TT1 to TT2 and TT2 to TT4, respectively. On the other hand, the value of  $\sigma_s$  increases, decreases and then sharply increases from specimens TL1 to TL2, TL2 to TL3 and TL3 to TL4, respectively. In contrast, the value of  $\sigma_s$  continuously increases from TT1 to TT4. The values of  $\sigma_0$  increases decreases and then sharply increases from specimens TL1 to TL2, TL2 to TL3 and TL3 to TL4, respectively. It is observed that the variation for  $\sigma_0$  is also same for T orientation samples. It can be noticed that the values of  $\sigma_{\rm S}$  and  $\sigma_0$  are always higher for fourth double aging cycle as compared to single and other two double aging cycles.

#### **IV. FRACTURE TOUGHNESS**

The plain strain fracture toughness ( $K_{IC}$ ) values of single and double aged starting materials are given in Table-5. Initially, all the specimens of starting materials were

aged followed by fatigue pre-cracking. After pre-cracking the specimens CL1 and LC1 were directly subjected to fracture toughness testing whereas the specimens CL2, LC2, CL3, LC3, CL4 and LC4 were re -aged as per cycle given in Table-5 and then subjected to fracture toughness testing. The variation in K<sub>IC</sub> values can be observed in Table-5. The K<sub>IC</sub> values of CL orientation specimens increases remain constant and then decreases again to initial values from CL1 to CL2, CL2 to CL3 and CL3 to CL4, respectively. On the other hand, the values of K<sub>IC</sub> continuously decrease from specimens LC1 to LC4. It is to be noticed that the specimens CL3 and LC3 have same KIC values of ~118 MPa√m. Similarly, the specimens CL4 and LC4 have same K<sub>IC</sub> value that is 113 MPa $\sqrt{m}$ . The anisotropy in fracture toughness is highest between CL1 and LC1 for single aged specimens. This anisotropy in fracture toughness decreases slightly after second aging of specimens to 150° C / 6Hrs / AC (CL2 and LC2). When second aging is performed at 250° C / 6Hrs / AC and 485° C / 6Hrs / AC it is observed that the CL and LC specimens display isotropic fracture toughness values.



#### Table-5. Fracture Toughness Values of Single and Double aged Starting Materials. Condition of Material

Condition of material [Initially all sam- ples solutionized at 820° C / 3Hrs /	Sample identifi- cation	Fracture toughness values (K <sub>IC</sub> ) (MPa√m)
AC		
195° C / 6Uma / A C	CL1	111.315
465 C/OHIS/AC	LC1	127.895
485° C / 6Hrs / AC +	CL2	117.96
Pre-cracking + Aging at 150° C / 6Hrs / AC	LC2	124.446
485° C / 6Hrs / AC +	CL3	117.661
Pre-cracking +Aging at 250° C / 6Hrs / AC	LC3	117.92
485° C / 6Hrs / AC +	CL4	112.69
Pre-cracking+Aging at 485° C / 6Hrs / AC	LC4	112.90

#### V. FRACTOGRAPHY

Scanning electron microscopic (SEM) images of fractured tensile test specimens which were subjected to single and double aging treatments have been shown in figure 11. All the SEM fractographs show concentric equiaxed ductile dimples of different sizes.

- (a) Single Aging at 485°C
- (b) Double Aging  $485^{\circ}$ C &  $150^{\circ}$ C
- (c) Double Aging 485°C & 250°C
- (d) Double Aging at 485°C

#### Longitudinal

#### Circumferential



Figure 11: SEM images of tensile fractured samples

#### **VI. CONCLUSIONS**

Following conclusions are made based on the results obtained from the present work:

The tensile flow curves for single and double aged specimens show best fit with Voce's model and the maximum strength is realized only after re-aging at 485°C / 6Hrs/ Air cooling.

The plain strain fracture toughness  $(K_{IC})$ values in longitudinal and tangential orientation of crack propagation display anisotropy for single aged starting material. The extent of anisotropy decreases as the temperature of second aging treatment during double aging. The increases optimum isotropy in fracture toughness is achieved at second aging temperature of 485°C. The reason for maximum isotropy in fracture toughness values by re-aging treatments at 485°C / 6 Hrs can be attributed to transformation of higher volume fraction of martensite precipitates during re-aging, which in turn completely eliminate the impression of flow lines formed during extrusion (or) prior deformation processes.

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