

# ULTRAHIGH STRENGTH, HIGH FRACTURE TOUGHNESS LOW-ALLOY STEEL: DMR-1700



Dr G Malakondaiah and Dr P Rama Rao

Defence Research & Development Organisation Ministry of Defence, New Delhi 110 011

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### ULTRAHIGH STRENGTH, HIGH FRACTURE TOUGHNESS LOW-ALLOY STEEL: DMR-1700

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### Preface

Ancient iron, the fourth most abundant element in the earth's crust, is an astonishing modern engineering material. Alloying behaviour of iron has been the subject of countless, exhaustive researches. Relations between yield strength and the microstructural features that arise when iron is alloyed and thermo-mechanically treated have firmly established the findings of the theory of strengthening mechanisms. Iron has thus served as a model material for metal sciences. Steels became the ubiquitous choice for structural applications and have come to be used almost anywhere and everywhere. Consequently billions of tonnes of steel are now produced annually. Among the over 3000 varieties of steel that have been developed and used, ultrahigh strength steels constitute an important group of speciality steels.

Several components of military and aerospace-related hardware require higher fracture toughness, for enhanced flaw tolerance, than hitherto attained in the known low-alloy ultrahigh strength steels. High-alloy maraging and secondary hardening steels provided the solution and these have been deployed in defence as well as in civilian applications that require higher fracture toughness at ultrahigh strength levels. The downside of these highly alloyed steels is that they are expensive. In this scenario, DMRL launched a major, prolonged research based development of a relatively low alloy, and therefore not-so-expensive steel.

Chapter 1 presents a review of the available literature on ultrahigh strength steels. A brief description of the basics of ductile fracture theory is given in Chapter 2. Recognising the gap in literature regarding the role of solute elements in determining the fracture toughness of dilute iron solid solutions, substantial original research effort to close this gap was undertaken. This is the subject matter of Chapter 3. Chapters 4 and 5 describe in detail DMRL work on the development (Chapter 4) and application (Chapter 5) of the indigenously developed ultrahigh strength high fracture toughness steel, designated DMR-1700 steel.

Summaries of the contents of Chapters have been given at the end of each Chapter.

Research based development of DMR-1700 steel, then pilotscale production and thereafter industrial-scale production, and finally its application has been a colossal programme lasting three decades. Numerous colleagues have contributed to this programme and they have been duly acknowledged in this compilation. Directors of DMRL have extended exemplary support without let-up, so also DMRL's sister laboratories as well as industrial houses. The credit for the success of this programme, if any, truly belongs to all these several individuals and institutions.

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We are grateful to many whose work has led to this compilation by us. Mentioned below are names of most of our colleagues; we are also thankful to those whom we may have missed mentioning here.

We are thankful to late Mr SLN Acharyulu, Dr D Banerjee, Dr AM Sriramamurty, Dr Amol A Gokhale, Dr SV Kamat, former Directors, and Dr Vikas Kumar, the present Director, DMRL for their interest in and support to the development of DMR-1700 steel as well as its industrial production and application.

We are grateful to the following senior functionaries for their enormous interest and help, but for which the development, production and application of DMRL steel could not have been accomplished.

Mr KK Sinha, Mr RK Mahapatra and Mr AK Taneja, Former CMDs of Midhani, provided leadership in establishing tonnage-scale production and supplying around 200 T of ring forgings and plates/ sheets of DMR-1700 steel.

Late Dr APJ Abdul Kalam, then Director, DRDL and Dr Prahlad, Project Director for their support to fabrication of DMR-1700 steel as one-to-one replacement for 18Ni1700 maraging steel for AKASH missile casings.

Mr RN Agarwal, then Director, ASL, spearheaded production of DMR-1700 steel for fabrication of AGNI missile motor casings.

As Chairman of Project Review Committee Dr A Subhananda Rao, then Director, Solid Propulsion Directorate, played an active role in progressing DMR-1700 steel production and fabrication of missile casings as well as development of coatings at CECRI. Late Dr Baldev Raj, then Director, Materials, Chemical & Reprocessing Group, later Director IGCAR, studied in-depth the failed missile casing and suggested solutions for fabrication, including welding, and ND examination.

Dr M Srinivas, Scientist DMRL has been part of the DMR-1700 steel development starting from its basic studies which he himself carried out. Mr J Marthanda Murthy, Scientist DMRL played a very helpful role in the development of DMR-1700 steel and its applications.

The scientists and officers of the Mechanical Behaviour Group (MBG) of DMRL played a very important role in the characterisation of mechanical properties during development, tonnage production and application of DMR-1700 steel. It was possible to generate voluminous test data because of these highly interested and committed officers.

We profusely thank Scientists Dr Vikas Kumar, Dr VK Varma, Dr SV Kamat and Dr N Eswara Prasad and Officers Mr E Durga Rao, Mr BB Sivakoti, Mr GB Vikram, Mr T Rajagopal Rao, Mr Ramadev and Mr P Ramaswamy for their interest and support.

Dr G Madhusudhan Reddy played an important role in applications of DMR-1700 steel, especially for AGNI missile casings and for base plate of 120 mm long-rage mortar.

MBG was supported by the Special Metals Group (SMG) in melting of the steel alloys, by the Analytical Chemistry Group (ACG) in analysing chemistry of the alloys, by the Structural Materials Group (SMG) in characterising the microstructure of the steel alloys and by the Metal Joining Group (MJG) in developing weld procedures.

Thanks are due to L&T for taking up fabrication of AKASH missile motor casings and to WIL for fabricating AGNI missile motor casings. Because of their keen interest and commitment, it was possible to establish fabrication technology for DMR-1700 steel. Similarly, fabrication of Base Plate for 120 mm Long Range Mortar was undertaken by Multifabs Engineering Contractors, Hyderabad.

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# List of Acronyms

AC	Air Cooled
Ac	Arrêt Chauffant
AE	Acoustic Emission
AES	Auger Electron Spectroscopy
AMS	Aeronautical Material Specification
AOD	Argon Oxygen Decarburization
Ar	Arrêt Refroidissant
ARDE	Armament Research & Development Establishment
ARIANE 5	Part of the Ariane Rocket Family
ASL	Advanced Systems Laboratory
ASTM	American Society for Testing and Materials
BHN	Brinell Hardness Number
BME	Ballistic Mass Efficiency
BMG	Bulk Metallic Glass
CC	Continuous Casting
ССТ	Continuous Cooling Transformation
CECRI	Central Electrochemical Research Institute
CHT	Conventional Heat Treatment
CIM	Contour Integral Method
CNC	Computer Numeric Controlled
СР	Commercially Pure
CRI	Chief Resident Inspector

СТ	Compact Tension
CVN	Charpy V-Notch
DBTT	Ductile-Brittle Transition Temperature
DC	Direct Current
DMRL	Defence Metallurgical Research Laboratory
DOP	Depth of Penetration
DP	Dual Phase
DP	Dye Penetrant
DRDL	Defence Research and Development Laboratory
DRDO	Defence Research and Development Organisation
DSA	Dynamic Strain Ageing
DSC	Differential Scanning Calorimetry
EAF	Electric Arc Furnace
EAM	Electric Arc Melting
ECAP	Equal Channel Angular Process
EIS	Electrochemical Impedance Spectroscopy
EPA	Epoxy Polyamide
EPMA	Electron Probe Micro-Analysis
ESR	Electroslag Refining
FC	Furnace Cooled
FEM	Finite Element Method
FP	Fluorescent Penetrant
GTA	Gas Tungsten Arc Welding
HE	Head-End
HRR	Hutchinson-Rice-Rosengren Zone
HTA	High Temperature Austenitising
HTAT	High-Temperature Austenitising Treatment
ICME	Integrated Computational Materials Engineering
ICV	Infantry Combat Vehicle
ID	Inner Diameter

### List of Acronyms

IF	Induction Furnace
IGCAR	Indira Gandhi Centre for Atomic Research
IIW	International Institute of Welding
Inco	The International Nickel Company
JMA	Johnson–Mehl–Avrami Theory
JSME Standard	Japan Society of Mechanical Engineers Standard
LOP	Lack of Penetration
LR	Ladle Refining
LRM	Long Range Mortar
LVDT	Linear Variable Differential Transformer
MBT	Main Battle Tank
MM	Mixed Microstructures
MSIP	Maraging Steel Indigenisation Programme
MWCNT	Multi-Walled Carbon Nano-Tubes
NDE	Non-Destructive Evaluation
NE	Nozzle-End
NF	No Failure
OCP	Open Circuit Potential
OD	Outer Diameter
PAGS	Prior Austenite Grain Size
PM	Parent Metal
PSLV	Polar Satellite Launch Vehicle
QT	Quenched and Tempered
RA	Retained Austenite
RHA	Rolled Homogenous Armour
RSP	Rourkela Steel Plant
RT	Radiography test
SAE International	Society of Automotive Engineers international
SBIR	Small Business Innovation Research
SCC	Stress Corrosion Cracking Resistance

### Ultrahigh Strength, High Fracture Toughness Low-alloy Steel: DMR-1700

SCE	Saturated Calomel Electrode
SEM	Scanning Electron Microscope
SIMS	Secondary Ion Mass Spectrometer
STITT	Short-Term Isothermal Transformation Treatments
SZW	Stretch Zone Width
SZWc	Critical Stretch Zone Width
TARB	Test Article Review Board
TE	Temper Embrittlement
TEM	Transmission Electron Microscopy
TIG	Tungsten Inert Gas
TME	Tempered Martensite Embrittlement
TR	Test Ring
TTT	Temperature-Time-Transformation
UHP	Ultrahigh Purity
UHS	Ultrahigh Strength Steels
USA	United States of America
UT	Ultrasonic Tests
UTS	Ultimate Tensile Strength
VAR	Vacuum Arc Remelting
VD	Vacuum Degassing
VHN	Vicker Hardness Number
VIM	Vacuum Induction Melting
VSSC	Vikram Sarabhai Space Centre
WBGB	Weld Bead Ground on Both Sides
WBGO	Weld Bead Ground on One Side
WIL	Walchandnagar Industries Ltd.
WPS	Specifications of Welding Procedure
XFEM	Extended Finite Element Method
XRF	X-Ray Fluorescence
YS	Yield Strength

# Symbols

a	Crack Length
a <sub>0</sub>	Initial radius of the void at the time of nucleation
A <sub>1</sub>	Boundary between the ferrite-cementite field and the fields containing austenite and ferrite or austenite and cementite
A <sub>3</sub>	Boundary between the ferrite-austenite and austenite fields
A <sub>cm</sub>	Boundary between the cementite-austenite and the austenite fields
A <sub>s</sub>	Austenite start temperature
b	Burger's vector
B2	CsCl crystal structure
bcc	Body center cubic
B <sub>s</sub>	Bainitic start temperature
d	Grain diameter
ds	Length increment along the contour
E <sub>nuc</sub>	Energy absorbed in the nucleation process
Egrow	Energy absorbed in the growth process
fcc	Face centered cubic
G	Strain energy release rate
HR <sub>c</sub>	Hardness Rockwell C scale
J	Parameter by Rice to characterise the crack tip

J <sub>IC</sub>	Critical J-integral value; suffix I stands for tensile mode of loading elastic-plastic fracture toughness
K	Stress intensity factor
K <sub>IC</sub>	Critical stress intensity factor; suffix I stands for tensile mode of loading plane strain fracture toughness or simply fracture toughness
K <sub>ISCC</sub>	Stress corrosion cracking resistance
K <sub>Q</sub>	Conditional value of fracture toughness
k,	Grain size exponent
l	Characteristic distance from the crack tip
M <sub>s</sub>	Martensite start temperature
n	Strain hardening exponent
Р	Applied force
Rc	Hardness Rockwell C scale
r <sub>p</sub>	Plastic zone size
T <sub>o</sub>	Thermodynamic limit to minimise the blocks of retained austenite
u	Displacement vector
V <sub>v</sub>	Volume fraction of austenite
V <sub>v-B</sub>	Volume fraction of blocky type austenite
$V_{\gamma-f}$	Volume fraction of the film type retained austenite
V <sub>b</sub>	Volume fraction of bainitic ferrite
ν	Poisson's ratio
σ	Stress
ρ	Dislocation density
λ	Interparticle distance
3	Transition carbide, called epsilon carbide
σε	Strain energy density
α	Martensite
γ′	Reverted austenite

Symbols

٤ <sub>av</sub>	Average plastic strain in the HRR zone exclusive of the
	process zone
$\sigma_{_{\rm f}}$	Critical fracture stress
ε <sub>f</sub>	Critical fracture strain
$\sigma_{_{\mathrm{flow}}}$	Local flow stress
$\Delta G^{o}_{B}$	Free energy of carbon segregation to grain boundaries
ΔΚ	Stress intensity range
$\sigma_{_{\rm m}}$	Hydrostatic stress (tensile)
ε <sub>n</sub>	Nucleation strain
$\sigma_{_{o}}$	Strength coefficient
$\gamma_{\rm p}$	Energy associated with the plastic deformation
$\gamma_s$	Energy associated with the surface energy
ε <sub>s</sub>	Lattice misfit strain
$\sigma_{_{ys}}$	Yield stress
$\sigma_{yt}$	Average of yield and ultimate tensile strength
Ī	Mean linear intercept grain size
$V_{\alpha_b}$	Maximum fraction of bainite that can form
X <sub>αb</sub>	Carbon that remains in the bainitic ferrite after the excess has been partitioned into the residual austenite
x	Average carbon concentration
$\delta_t$	Crack tip opening displacement
$\delta_{tc}$	Critical crack tip opening displacement
θ	Angle from the plane of the crack

### **CHAPTER 1**

### **Ultrahigh Strength Steels – A Review**

### **1.1 INTRODUCTION TO STEEL AS AN ENGINEERING MATERIAL**

Steels are the material of interest in our work. Iron and steel have indeed been researched more extensively than any other metallic material and, overtime, more than 3000 varieties of steel have been developed and used in making a 'paper clip to an aircraft carrier'. India is the third largest producer of steel having produced this year 101 million tonnes of finished steel, behind only Japan and China.

From the perspective of strengthening mechanisms, iron serves as an outstanding model material. Strengthening can be achieved by several means as shown<sup>1</sup> in Table 1.1. These strengthening mechanisms have made possible increasing strength from as low as about 200 MPa of ultrahigh purity iron to as high a strength level of 4000 MPa in

Mechanism	Relation
Dislocations	$\Delta \sigma \approx \alpha \ \mathrm{Gb} \sqrt{\rho}$
Grain size	$\Delta \sigma \approx \Delta \sigma_0 + k/d^{1/2}$
Bainitic ferrite plate size	$\Delta \sigma \approx 115 (\overline{L})^{-1}$
Solid solution	$\Delta \sigma \approx A (\varepsilon_s)^{3/2} C^{1/2}$
Precipitation and dispersion	$\Delta \sigma \approx \beta Gb/\lambda$
Multiphase	$\Delta \sigma \approx f(\sigma_{II}, \sigma_{I})$

 Table 1.1. Strengthening mechanisms.

G = shear modulus;  $\rho$  = dislocation density; b = Burger's vector;

d = grain size;  $\overline{L}$  = plate size; C = concentration of solute;  $\lambda$  = interparticle distance;

 $\sigma$  = yield strength;  $\varepsilon_s$  = lattice misfit strain; A,  $\beta$  = constants

Fe-based bulk metallic glass (Fig. 1.1, Table 1.2). As relevant data for reference, dislocation free iron whisker shows a strength of 13,000 MPa; recently, after the discovery of carbon nanotubes, multi-walled carbon nanotubes have been made with a strength level of 63,000 MPa, which far exceeds the theoretical strength of 27,300 MPa.

### **1.2 ULTRAHIGH STRENGTH (UHS) STEELS**

Broadly, there are four classes of Ultrahigh Strength (UHS) steels: (a) low-alloy steels typified by AISI 4340 developed during the 1940s, (b) high-alloy maraging steels typified by 18Ni1700 steel developed during the 1960s, (c) high-alloy secondary hardening steels typified by AF1410 developed during the 1970s, and (d) carbide-free bainitic steels developed during the 1980s. These four classes of UHS steels,



#### Abbreviations:

1. UHP - Ultrahigh Purity 4. ECAP - Equal Channel Angular Process 2. CP - Commercially Pure 5. UHS - Ultrahigh Strength 3. DP - Dual Phase, Industrially Cold Rolled 6. BMG - Bulk Metallic Glass

7. MWCNT - Multi-Walled Carbon Nano-Tubes



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#### About the Book

Steels for several high-technology applications, such as military hardware and aerospace, need to possess ultrahigh strength (UHS; minimum yield strength of 1380 MPa (200 ksi)) coupled with high fracture toughness in order to meet the requirement of minimum weight while ensuring high reliability. Broadly, there are four classes of UHS steels: (a) low-alloy steels typified by AISI 4340, (b) high-alloy maraging steels typified by 18Ni1700 steel, (c) high-alloy secondary hardening steels typified by AF1410, and (d) carbide-free bainitic steels. Distinctive characteristics of the UHS low-alloy, maraging, secondary hardening and bainitic steels have been presented in the monograph. In such applications, though performance of materials is of prime consideration, cost and availability make low-alloy steels an attractive option. However, the use of low-alloy steels has often been limited by their low fracture toughness.

A comprehensive research programme was launched at the Defence Metallurgical Research Laboratory (DMRL) during mid-1980s to develop a cost effective low-alloy alternative to the highly alloyed 18Ni1700 maraging steel. The monograph describes the more than three decades of R&D work that led to the researchbased development of a new UHS low-alloy steel, designated DMR-1700, its production and its defence applications. The strength and toughness combination of DMR-1700 steel is better than the best reported values of low-alloy steels AISI 4340, 300M and D6ac, and is comparable to that of 18Ni1700 maraging steel. Given this result, DMR-1700 steel has been successfully demonstrated for (a) one-to-one replacement of maraging steel for missile casings, (b) substantial reduction of weight of the base plate for 120 mm long range mortar and (c) superior ballistic performance for armour applications. To address cost effectiveness of DMR-1700, DMRL pursued ladle refining and vacuum degassing + continuous casting route and found that the steel met the chemistry and properties successfully.

#### **About the Authors**

**Dr G Malakondaiah** obtained his PhD in Metallurgical Engineering from Banaras Hindu University (BHU) in 1980. He served on the Metallurgy faculty of BHU for two years as lecturer (1980-82) prior to his joining Defence Metallurgical Research Laboratory (DMRL) in October, 1982. His research work has been broadly in the area of advanced mechanical metallurgy, with emphasis on structure-mechanical property correlations in metals and alloys. He has been interested in the development of specialty steels. Thus he got engaged in the comprehensive research programme aimed at developing a cost effective low-alloy alternative to the highly alloyed 18Ni1700 maraging steel. This major programme, launched during the mid-1980s, eventually led to the development of DMR-1700 steel. In recognition of his contributions to specialty steels, including DMR-1700 and naval steels, he was awarded Agni Award for Excellence in Self-Reliance (2005) by DRDO and DRDO Technology Leadership Award-2010. He retired from DRDO, as Distinguished Scientist, in 2014 after serving as Director of DMRL (2007-2013) and subsequently as Chief Controller R&D of DRDO (2013-2014). Post-retirement from DRDO, he also served as Steel Chair Professor at NIT-Warangal (2015-2016). He is a Fellow of the Indian National Academy of Engineering and the Telangana Academy of Sciences.

**Dr P Rama Rao** is at present Chairman, Council of Indian Institute of Science (IISc), Bangalore and International Advanced Research Centre for New Materials, Hyderabad. He was a Professor of Physical Metallurgy at Banaras Hindu University and he has served as Director, Defence Metallurgical Research Laboratory, Hyderabad, as Distinguished Scientist in Defence Research and Development Organisation, as Secretary to Government of India, Dept of Science and Technology, as Chairman, Atomic Energy Regulatory Board and then as Vice-Chancellor, University of Hyderabad. He was awarded distinguished Professorship by Indian Space Research Organisation (ISRO). He is a Member of the Atomic Energy Commission. He has been elected Fellow of The Royal Academy of Engineering (UK) and Foreign Member of the US National Academy of Engineering. He has been President, Indian Academy of Sciences (1995-97), President, Indian National Academy of Engineering (2001-03), General President, Indian Science Congress Association (1997-98), and President, Indian Nuclear Society. He was President, International Congress on Fracture (1989-93) and Vice-President, International Union of Materials Research Societies (2002-03). He was a member of the Science Advisory Council to the Prime Minister and a member of the National Security Advisory Board. He was awarded Padma Vibhushan (the second highest civilian award) by the President of India in 2011.

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