

MARAGING STEEL : LAB TO LAUNCH

A RECOLLECTION

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By

P P Sinha

MATERIALS PROCESSING DIVISION
MATERIALS AND METALLURGY GROUP
VIKRAM SARABHAI SPACE CENTRE
TRIVANDRUM

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Dear Fellow Members:

The release of this issue of "met News", coincides with the launch of India's first Polar Satellite Launch Vehicle (PSLV). Although, the launch has not been fully successful, it is a matter of great satisfaction to technical communities of the country that all the subsystems of PSLV have performed extremely well. Particularly, a number of indigenously developed materials in these systems have proven their flight worthiness. It is, therefore, highly opportune that the Editorial Committee has decided to publish, in quick succession, the experiences of some of its members on the indigenous development of various metallic materials for the PSLV. In this series, I have been asked first to present my experience of over two decades on the indigenous development of maraging steel for Indian space programmes. I am indeed thankful to the Committee for this gesture.

Development of maraging steel for PSLV is, in true sense, one of the greatest national achievements. Through this recollection, I would only like to highlight the various efforts that have gone into making indigenous maraging steel within ISRO and at other national organisations.

Looking back, I joined Vikram Sarabhai Space Centre (VSSC), ISRO on the 6th December 1971, after my Masters Degree in Metallurgy from the Department of Metallurgical Engineering, Banaras Hindu University. The first task assigned to me was the development of "maraging steels" by the then Head, Materials Quality Control Division - Dr. M.K. Mukherjee. For a while I was totally taken aback! For, I was given the task of developing a material about which I

knew nothing, except that the name sounded a bit familiar. Incidentally, within a week of my joining I had to go to my native place (Muzaffarpur, Bihar). There, one day when I was casually going through some of my old undergraduate class notes, I suddenly noticed that Prof. T. R. Anantharaman, the then Head, Department of Metallurgical Engineering at BHU, had

given the definitions of "maraging" and "ausageing" in one of his lectures on "Ultra-high strength steels". I must admit, that was my first acquaintance with Maraging Steel.

What is Maraging Steel?

Maraging Steels are primarily based on two solid state reactions: Martensitic transformation in Fe-Ni matrix and its subsequent ageing in presence of other alloying elements. Fe-Ni martensite soft and BCC in crystal structure compared to the Fe-C martensite which is B.C.T. in structure, hard and brittle. In presence of alloying elements like Co, Mo and Ti, the Fe-Ni martensite, on simple ageing at 753K for 180 minutes, is strengthened by the fine distribution of N_3Mo , Ni_3Ti and $6-Fe-Mo$ precipitates on dislocations. The material is of special significance to the aerospace designers because of its high strength (say, for 250 grade, UTS of 1800 MPa) coupled with high fracture toughness ($KIc = 100 \text{ MPa}\sqrt{\text{m}}$), simple heat Treatment, negligible dimensional distortion, easy fabricability including high weld efficiency.

Early Phase of indigenous Development

As usual for any developmental activity, an intense literature survey was undertaken. It was soon realised that although extensive work on the steel had been done during 1960-70 on international level there was hardly any concrete work in India except that of Prof. A. K. Seal and his Colleagues (B.E. College, Shibpur) and Dr. Rajendra Kumar et. al (National Metallurgical Laboratory, Jamshedpur) on the ageing behaviour of Mn and Cu containing maraging steels.

Work at VSSC on Japanese Steel:

At Vikram Sarabhai Space Centre (VSSC), ISRO, under the leadership of Dr. K. V. Nagarajan (presently Head, Materials and Metallurgy Group), I started work on sample level from a maraging steel rolled piece of around 300 x 300 x 8mm brought by Dr. M. K. Mukherjee from Japan during one of his earlier visits. A detailed study on the influences of heat treatment variables on strength and microstructure were carried out. Effects of plastic deformation on the ageing behaviour of the steel were also investigated and was presented to the IIM, ATM 1993 held at BHU, Varanasi.

350 Grade Steel Shattered!

We started making this steel in a button melting furnace under argon atmosphere. I distinctly remember the day in the summer of 1973, when we melted 350 grade (These grades are defined on the basis of yield strength in KSI) maraging steel. On getting visually a good button of around 25mm diameter, I was so thrilled that I rushed to Prof. T. R. Ramachandran (presently Director, Jawaharlal Nehru Centre for Aluminium, Nagpur) who had come from IIT, Kanpur to work on TEM and proudly claimed that I had made 350 grade maraging steel. Prof. Ramachandran in his usual cool asked me, Sinha, should I break it? We were all taken aback when the piece broke into pieces on dropping on the floor!. I must admit that the incident had severe impact on me for some time. However, the challenge as I realised later, turned out as a great force in me to face many more odds during the later stages of developments.

TISCO Contacted:

R & D Unit of Tata Iron and Steel Co (TISCO) was approached to make maraging steel in early 1974. It must be acknowledged that TISCO could make the steel on experimental basis and supplied a piece of 150mm long and 50mm diameter rod for evaluation. However, since they did not have vacuum melting furnaces, the project could not be pursued further.

Laboratory Scale Production at VSSC

Our melting furnaces (AIM & VAR), forging press and rolling mill were

commissioned by the end of 1974. Once these facilities were established, development started in full swing on 250 grade steel. We would establish various processing parameters like melting, forging, rolling and heat treatment by 1976. During the same period, when MIDHANI was still in its infancy, we also made a number of semi finished products from the steel melted in VSSC for use in launch vehicle programmes. These included, (a) forged blocks and cold-rolled strips of around 3000mm long, 150mm width and 1.0mm thick (b) wires of 0.1mm to 1.0mm in diameter using the wire drawing facility of AFD, BARC and © Fasteners by thread rolling at HMT, Kalamassery:

Stainless Maraging Developed!

The period 1977-79 witnessed another significant milestone when a new variety of 250 grade stainless maraging steel was developed at VSSC. It is heartening to note that this steel was later christened as "INDIAN MARAGING STEEL" by none else than Prof. R. F. Decker -the father of 18 Ni-Co variety maraging steel.

Development of Maraging Steel version of RH-300 Motor Case

M/s. MIDHANI backed up with French Know-how started melting steel sometimes during 1979-80 on tonnage level. Commissioning of MIDHANI facilities gave big boost to the maraging steel team at VSSC to take up activities on rocket motor case fabrication.

The first attempt in this regard was to make a RH-300 version motor case (300mm in diameter and 2.0m in length) under the leadership of Shri S. Arumugham. Using the cold rolled sheets of 1.5mm thick imported from M/s. Aubert and Duval, France and forged rings of 300mm diameter from MIDHANI, the fabrication was carried out at Mechanical Engineering Facility (MEF) of VSSC. The motor case was tested to burst during early 1982.

Material Selection for PSLV Booster case:

In all previous launch vehicle programmes of ISRO, a low alloy high

strength alloy, 15 CDV6 steel had been used as motor casing. Even the project PSLV envisaged usage of 15 CDV6 steel as its booster case material. Enthused by excellent properties and immense data generated at VSSC, this steel was proposed as a candidate material for the PSLV motor case by the maraging steel developmental team. Considering this merit of the proposal, the Chairman, ISRO, Prof. Dhawan constituted a National level committee in 1981 under the Chairmanship of Late Dr. Brahma Prakash to select material for the PSLV booster case. This Committee unanimously recommended the use of 250 grade maraging steel. However, in its far fetched recommendation it also suggested that ISRO should take up immediate action on the indigenisation of the steel.

Maraging Steel Indigenisation Programme (MSIP):

In January 1982, a Maraging Steel Indigenisation Programme (MSIP) was formed under the directorship of Dr. B.K.Sarkar (presently Director, CSCRI, Calcutta). Considering the massive nature of the task, a working level project team identifying me as Project Leader was constituted by the Director, MSIP. In order to accomplish the task within the stipulated time the team initiated parallel action to generate detailed material specifications, get the material melted and rolled into plates and rings on the larger scale utilising the national facilities, evolve techniques for microstructural characterisation, fracture toughness evaluation and NDT. Detailed studies were also taken to establish welding parameters.

Material Realisation

One of the serious problems that emerged during the initial phase of indigenisation was MIDHANI'S submission to Dr. Brahm Prakash Committee that they cannot make maraging steel ingot larger than 400mm in diameter and 2.5 tons in weight. Since, ingots of minimum 5-6 tons and 700mm diameter was required, I was deputed to MIDHANI and to find out the ways. I met Prof. Gude, my erstwhile teacher at BHU and Chief of Technology Group at MIDHANI those days. During the course of discussion I came to know that they had VAR furnace

capable of melting around 4.0 tons of Ti-alloys. I immediately asked Prof. Gude, why can't we use this furnace for maraging steel and realise the desired size ingots? I was told that the primary ingots obtained through VIM would be only of 2.5 tons and maximum of 400mm diameter. Even if they join three ingots as per my later suggestion, they cannot melt 400 diameter electrode in 800 diameter crucible. Not to leave the opportunity, I asked why not melt the 400 diameter joined electrodes first in 600mm diameter crucible and then go for second remelting in 800 diameter. This proposition however, met with serious objection on the ground of high cost. To me, nevertheless, it was not a deterrent factor as I was more than convinced that the double VAR was advantageous because i) it would ensure full homogeneity of the melt ii) improve cleanliness specially with respect gases and iii) would pave the way for realising material through indigenous source. Probably, convinced with my argument, Prof. Gude assured me that they would examine these possibilities. We were happy to note that MIDHANI soon confirmed their willingness to supply maraging steel blooms of desired weight and sizes by adopting double VAR technique. This was the beginning for the INDIGENISATION OF MARAGING STEEL for PSLV.

Steel Making

It is to the credit of MIDHANI that they could make the steel of international standard within short span of time. Slabs and blooms were forged, tested and qualified jointly by MIDHANI and VSSC.

Plate Rolling

Initially, Rourkela Steel Plant (RSP), Rourkela showed serious reservations on taking up the work because of high strength of the steel, requirements of controlled atmosphere (with respect to Sulphur and oxygen) in reheating furnace, strict control over annealing temperature and thickness tolerances etc.

However, with greater persuasions and detailed technical input on rolling aspects of the steel provided by VSSC, RSP finally agreed to take up rolling on developmental basis more as national endeavour rather than a commercial proposition. It must be acknowledged that the bold decision taken by the Managing Director, RSP, Late S/Shri V. Subramony and Shri Subrata Ray (then DGM Mills) played key role in the indigenisation effort. The rolling of maraging steel plates of such large size (10m x 1.5m x 7.8mm) with specified dimensional tolerances, mechanical and metallurgical properties at RSP was a rare feat even on international level !

Ring Rolling

Rolling of rings was initially taken up at M/s. Echjay Steel, Rajkot. However, because of constraints of the mill capacity, the rings of 3.0m size could not be realised indigenously. MIDHANI contacted M/s. Krupp Steel, Germany for rolling rings from the bloom made at MIDHANI. Although M/s. Krupp Steel, agreed for the work, they conceded that they did not have any expertise on maraging steel. Based on the confidence generated at VSSC and MIDHANI, this programme was also taken up on a joint venture. S/Shri VK Gupta from MIDHANI and S. Arora and myself from VSSC were deputed to M/s. Krupp for rolling two rings of 3.0m in diameter. To our great satisfaction two rings of desired sizes and mechanical properties could be rolled in close co-ordination with M/s. Krupp Steel.

Technology Development Programme

While the material realisation efforts were continuing at M/s. MIDHANI and other places, a massive technology establishment programme was undertaken in the Materials Group of VSSC. These included microstructural characterisation by K. Sreekumar, A. Natarajan and myself, fracture characterisation by V.Divakar, S. Arumugham and Dr. T. S. Lakshmanan and welding technology development by a

group of engineers lead by Shri B. R. Ghosh. A separate team was also identified to evolve NDT procedures. These programmes resulted in generation of data base for the evaluation of materials and fabricated components at the later stages of product realisation.

MSIP Concluded:

Maraging Steel Indigenisation Programme was successfully completed by the end of 1983. A detailed report covering all the development aspect were made for future reference and guidelines. It is no exaggeration that with this successful event, the country had demonstrated its capability of mastering technology of Maraging Steel.

Production Phase:

On successful completion of MSIP, MIDHANI was awarded the contract for the bulk requirements of plates, rings and filler wire for PSLV. While the plates could be rolled following the earlier established parameters without much problem (except few minor changes incorporated in hot levelling stage and also introduction of surface grinding at M/s. EOS, Madras to facilitate reliable UST), the first batch of 16 rings rolled at M/s. Krupp Steel faced possibility of rejection due to low fracture toughness ($65 \text{ MPa}\sqrt{\text{m}}$ - $80 \text{ MPa}\sqrt{\text{m}}$ against a minimum of $90 \text{ MPa}\sqrt{\text{m}}$ required). Extensive microstructural analysis carried out both at VSSC and MIDHANI revealed that the deterioration in toughness was primarily due to thermal embrittlement. On receipt of procedural details from Krupp Steel, it was found that the embrittlement was due to some critical process deviations.

Development of Heat Treatment Programme to Salvage Rings!

Because of high cost and strategic nature of the material, both MIDHANI and VSSC agreed to find out the ways to salvage these rings. This resulted in series of experiments at both places. By that time, Prof. P. Rama Rao (presently Secretary, Department of Science and Technology, Govt. Of India) had joined Defence Metallurgical Laboratory (DMRL) as Director. MIDHANI proposed to send a team of experts to M/s. Krupp, Germany under the leadership of Prof. Rao

to (I) evolve salvage heat treatment procedures for the embrittled rings and (ii) firm up the processing schedules for the rolling of subsequent batches. The proposal was readily

agreed by VSSC and a team consisting of Dr. Nageswar Rao from MIDHANI, Shri Kalyanasundaram (presently at KELTECH) and myself from VSSC and Prof. Rama Rao from DMRL were deputed to Krupp Steel, Germany for the purpose.

As a person responsible from VSSC, ISRO team to present complete metallurgical analysis on as the acceptance of these rings, inclusion of Prof. Rama Rao as team leader came as a great relief to me. I had the privilege of being his student at BHU and had known his immense technical capabilities and humanitarian considerations was sure that in all my analysis, I would be properly guided by him. Although, maraging steel had not been an active field of Prof. Rao's research, true to our expectations, he went through the literature submitted to him very methodically and discussed at length all the data generated at VSSC and MIDHANI. It was a matter of great satisfaction that the salvage heat treatment evolved by the team under his guidance could restore the toughness of the rings.

Finalisation of Ring Rolling Programme

For future batches of rolling, all the previous data were analysed and a detailed schedule was worked out. However, some inconsistencies continued to persist with respect to toughness. It was to the credit of Shri V K Gupta and team at MIDHANI who suggested post rolling annealing at 950 °C before going for regular solution treatment. This again sparked off numerous experimental trials both at MIDHANI and VSSC to validate the proposal. Analysis of the results boosted the confidence of team to go in for an intermediate annealing operations. The decision was jointly endorsed by MIDHANI, VSSC and Krupp Steel in the beginning of 1986. I must confess at this stage that from reliability considerations, I was not very much convinced that the intermediate annealing

was adequate for one type of ring which had maximum cross section. Fortunately, my concern was eliminated during subsequent batches of rolling except in one case when one of the rings of this class showed problem due to inadequate working and had to be salvaged by cutting out the defective portion.

FABRICATION OF MOTOR CASES:

Once the plates and rings were realised and welding parameters were evolved during MSIP, other critical aspect of the programme was to develop fabrication technology on industrial scale. Initially, BHEL, Hyderabad carried out extensive work on roll bending and welding of one segment (cylindrical shell of 2.8m dia and 1.5 length). However, subsequent analysis revealed that a lot more were needed to be improved on fabrication aspect. Meanwhile M/s. L & T, Bombay and WIL, Walchandnagar were given contract to make motor cases for PSLV. These agencies also carried out number of welding studies.

Development of a New Filler Wire

One major problem that arose concerning welding was that the weldments had lower K_{ic} than the design requirements. This created a serious impasse on the progress of fabrication of motor case. Extensive microstructural analysis of the welds by S/Shri K. Sreekumar, A. Natarajan and myself at VSSC revealed that there was a need to change the filler composition to meet the critical design requirements of at least 90% weld efficiency over both tensile and fracture toughness. I was directly involved in designing a modified version of filler wire which was later validated by conducting extensive welding studies by Shri M. S. Kurup and colleagues at MEF, VSSC. Dr. Gururaja and team at BHPV, Vizag and L & T, Bombay. It is a matter of great satisfaction to me that the filler wire composition, thus developed, is being used for all welded construction of maraging steel in ISRO and Defence Programme.

Other Developmental Activities

A number of other technical problems also cropped up while fabricating motor cases and other sub systems of maraging steels. These problems were solved by systematic experimental analysis at VSSC and subsequent corrective actions at work centres with active participation of the fabricating agencies.

Some typical examples are:

- i) Analysis of heat affected Zone-II / parent metal interface cracking and suggestions for remedial action
- ii) Development of repair weld and local ageing parameters for the welded, machined and fully aged segments
- iii) Development of detailed process parameters for high duty fasteners and merman bands
- iv) Extensive studies on stress corrosion and hydrogen embrittlement etc
- v) Development of the 'Insitu' electro polishing device for non-destructive metallography analysis for quality and process parameter optimisation

CONCLUSION

The development programmes on maraging steel from laboratory scale at VSSC to the fabrication of PSLV booster case involving number of external agencies concluded with successful proof pressure test of number of motor cases (a real test for the material) and subsequent static test of first stage of PSLV in October 1991. The latest launch of PSLV has further confirmed the excellent performance of indigenously developed maraging steel.

P P SINHA
MPD, VSSC

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