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Erosion Resistance of Chromium–Manganese Iron Alloy Cast in Metal and Sand Moulds: PLS and DBAR Studies

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The wear-resistant high chromium (Cr: 16–19 %) iron alloyed with 5 % and 10 % manganese (Mn) was produced in metal and sand moulds by induction melting technique. The erosion resistance, hardness and microstructure were evaluated both in the as-cast and heat, treated conditions. The advanced non-destructive test (NDT) methods, namely Positron Lifetime Spectroscopy (PLS) and Doppler Broadening annihilation radiation (DBAR) studies, using variable energy positron beam were made use of to study the influence of metallurgical parameters on the defect sensitivity in the bulk and surface of the alloy. The data reveals that as the mould type is changed from metal to sand, the hardness decreases irrespective of the sample condition (i.e. as-cast or heat treated), whereas the erosion volume loss shows an increasing trend. The light and scanning electron microscopies give good support to these data findings. It is observed that faster the cooling rate (metal mould), finer is the carbide size precipitation on the surface of the sample. The PLS data reveals that the defect size and its concentration are higher for sand mould alloy compared to metal mould. Reasons for lower erosion loss and fewer defects of smaller sizes in metal mould are attributed to faster heat transfer in the metal mould compared to the sand mould. Further, heat treatment of the samples yielded spherodization of carbides in the matrix and some of the defects seem to have been annealed out leaving only fewer defects of smaller size in the alloy. The S-parameter profiles of 10 % Mn both in AC and HT samples are almost identical indicating near absence of any modification of defect structure near the surface following heat treatment in 10 % Mn sample, while 5 % Mn samples exhibit less defect concentration both at the surface as well as in bulk which agrees with the PLS results. Hence, the 5 % Mn bearing metal mould sample in the heat-treated condition is preferred choice as it shows higher hardness, lower erosion loss as well as least defect concentration with smaller defect sizes. Based on this investigation, a good correlation among erosion loss, DBA and PLS data has emerged.

Keywords: *Cr–Mn cast iron, Metal mould, Sand mould, As-cast, Heat treatment, Hardness, Erosion, Positron lifetime spectroscopy, Doppler broadening spectroscopy.*

1.0 INTRODUCTION

The wear and erosion of critical parts, such as coal and ash handling equipments, pressure parts of thermal power generation, result in metal wastage on account of high percentage of alpha quartz [1] present in coal causing wear damage. Therefore, several attempts have been made to withstand wear and erosion problems. The material which has been found to be a promising

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wear-resistant material is high chromium (Cr) iron [2] as contains hard carbides (M_7C_3) in a martensitic matrix, but fails to withstand sudden load/shock. To improve the impact resistance, manganese (Mn) is added, as Mn is an austenite stabilizing agent. The investigations reported [3] on the Mn addition up to 4.4 % in chromium iron has shown higher toughness compared to high chromium white alloy irons. Further, the use of Mn beyond 4.4 % in such alloy system is less reported. Hence, the present work reports on the erosion behavior of Cr (16–19 %)–Mn (at 5 % and 10 %) iron cast in metal and sand moulds affecting the erosion characteristics both in the as-cast and heat-treated conditions. The optical microscopy has been employed to look into the microstructural features erosion behavior with respect to the material condition. Also, positron lifetime spectroscopy and Doppler broadening annihilation radiation (DBAR) have been used to study the defect morphology in terms of defect concentration and size at the surface as well as the bulk to understand the influence change in mould type on Cr–Mn iron samples both in the as-cast and heat-treated conditions, as they have bearing on erosion loss from the surface and its connection to bulk nature.

The bulk positron life time spectroscopy technique has established a good correlation between fatigue life ratios and PLS parameters [4,5] in stainless steels for prediction of early fatigue damage. The work related to correlation of erosion behavior with surface defect characteristics in Cr–Mn alloy systems does not seem to exist in literature. Hence, the usefulness of DBAR in integration with PLS from the point of defect quantification with respect to hardness and erosion behavior involving microstructure in 5 % and 10 % Cr–Mn iron samples produced in sand and metal moulds is reported in this work.

2.0 EXPERIMENTAL PROCEDURES

The metal and sand moulded test samples of size 75 × 25 × 6 mm^3 were given austenitization soak at 960ºC for 2 hours followed by oil quenching and then finally tempering at 200ºC for 30 min with air cooling to room temperature. The as-cast and heat-treated samples were subjected to hardness measurements, microstructural examination using optical and scanning electron microscopy (SEM) and defect characterization using slow positron beam spectroscopy and conventional positron lifetime spectroscopy. The details of the melting and casting procedures of Cr–Mn alloy system under investigation are covered in detail in our earlier published work [6].

2.1 Erosion Test

The erosion test procedure consists of the flow of silica sand abrasive particles (AFS 60 grade) along with moisture-free compressed air at a particle velocity of 35±2 m/s and making an impact (impact angle of 45°±2°) on the target sample through a ceramic nozzle of diameter 5 mm for 30±2 seconds. Prior to this, the initial weight of the sample is measured using a digital electronic balance. The mass flow rate (m) of abrasive particles is maintained at 600–700 g/minute. The sample is then cleaned at the end of the test (30 s: test duration, i.e. t) and the final weight of the sample is measured in the same balance. The difference in weight (w) between the initial and final readings gives the measure of erosion loss of the sample. The erosion volume per kg, i.e. cc/kg is calculated using the formula $E_v = w \cdot m/d \cdot t$, where d is the density of Cr–Mn iron sample (7.8 g/cc).

2.2 Hardness and Other Measurements

The hardness measurement in terms of Rockwell ‘C’ scale (HRc) is done using a diamond cone type of indenter, at a load of 150 kg and average of six readings taken on two representative samples of the same history is reported. The microstructural features of the test samples examined using a light microscope are presented. The retained austenite (RA) content (%) of the sample is measured using X-ray stress analyzer equipment. The eroded surface features of the samples are done using SEM.

2.3 Slow Positron Doppler Broadening Annihilation Radiation, Measurement (DBAR)

DBAR measurement was carried out in Cr–Mn iron alloy prepared as described in metal and sand moulds and in as-cast and heat-treated conditions using the slow positron beam. The present beam has three components interconnected under high vacuum viz. (i) slow positron production (source and moderator), (ii) focusing and transport (Einzel lens and magnetic transport) and (iii) acceleration of positrons at the target. Positrons emitted from a sealed ^{22}Na source are moderated by 1-μ thick tungsten single crystal. The thermalized positrons come to the surface as tungsten has negative work function for positrons. The positrons extracted from the moderator are focused by the Einzel lens and are guided towards the sample through a magnetically guided assembly (90° bent solenoid and two Helmholtz coils). The positron energy varied by floating the sample at different voltages. The energy range of the positron beam is 200 eV–50 keV. Doppler broadened annihilation radiation measurements were carried out using an HPGe detector having resolution of 1.7 keV at 1332 keV photo peak of ^{60}Co. A spectrum with 10^6 counts was acquired at each energy. The shape parameter, namely, *S*-parameter defined as the ratio of the number of counts falling in a fixed energy window (±1 keV) centered at 511 keV to the total number of counts under the Gaussian peak, was evaluated. The *S*-parameter at the surface and in bulk was determined using computer program VEPFIT. The variation in the *S*-parameter as a function of depth gives the defect depth profile in the sample.

2.4 Positron Lifetime Spectroscopy (PLS)

Positron annihilation lifetime spectra were recorded at room temperatures in the Cr–Mn iron system using a fast-fast coincidence system with BaF_2 scintillators coupled with photo multiplier tubes and quartz window as detectors in about 1–2 hours. Three Gaussian time resolution functions were used in the lifetime analysis for fast and good convergence keeping the net resolution function around 220×10^{-12}s. The details of the experimental procedure and analysis can be found in our earlier work [7]. All spectra were analyzed into two lifetime components with the help of the computer program PATFIT-88 [8] with proper source and background corrections. The first lifetime component is called the bulk lifetime (also called free lifetime, $\tau_1 = \tau_b$), and the second lifetime is called the defect-state lifetime ($\tau_2 = \tau_d$) with respective intensities I_1 and I_2. In the present analysis, τ_1 is fixed at 107 ps which corresponds to lifetime of positrons in Fe and in the present systems Fe is the matrix. The fixed analysis will not suppress any information because the free annihilation lifetime does not provide any material information. The trapping rate (k), which is a measure of the defect concentration in the system, is estimated adopting the two-state trapping model [9]. Parameters of the trapping model are the positron lifetimes in the free and trapped states with intensities I_1 and I_2. The rate at which transitions from the delocalized states to the localized ones happen is the trapping rate. This transition rate (positron trapping rate, k) is proportional to the concentration of the defects. The τ_d is larger than τ_b for open-volume defects, such as vacancies, dislocations due to the decreased electron density in the defect site compared to the bulk material. The average positron lifetime is evaluated using experimentally obtained lifetimes

$$\tau_{av} = I_1\,\tau_1 + I_2\,\tau_2 \text{ (with } \tau_2 = \tau_d) \qquad (1)$$

Then, the trapping rate k is calculated from the equation below.

$$C_d = \frac{k}{\mu} = \frac{1}{\mu\tau_b}\frac{\tau_{av} - \tau_b}{\tau_d - \tau_{av}} \qquad (2)$$

The proportional constant μ is the trapping coefficient, and is obtained by an independent reference method.

3.0 RESULTS AND DISCUSSION

The chemical analysis data carried out using optical emission technique for Cr–Mn samples are shown in Table 1. Results in respect of erosion, hardness, retained austenite (% RA) and carbide volume (CV %) are shown in Table 2, and the slow positron lifetime data both for as-cast (AC) and heat-treated (HT) conditions are given in Table 3. The sample designation followed in Table 1 is as follows: first numeral (% Mn) followed by a letter (mould type) and lastly a number (section size).

TABLE 1

CHEMICAL COMPOSITION ANALYZED

Sample designation	Type of casting	Section size (mm)	Composition (wt. %)							
			C	Mn	Cr	Ni	Mo	Si	P	S
5M24	PM	24	2.55	4.6	18.40	0.95	1.70	2.15	0.090	0.044
5S24	SM	24	2.95	4.5	18.00	1.10	1.70	1.80	0.062	0.037
10M24	PM	24	2.75	9.6	16.70	0.90	1.65	1.90	0.090	0.040
10S24	SM	24	2.70	9.5	16.10	1.00	1.60	1.90	0.085	0.030

TABLE 2

DATA ON EROSION LOSS, HARDNESS, RA (%) AND CV (%) OF Cr–Mn IRON SAMPLES

Sl. No.	Sample designation	Hardness HRc(Av) (±1)	RA %		CV %	Erosion volume loss (cc/kg × 10^{-3})
			Range	(Av)	Range	
1	5M24 AC	55	56–58	57	26 – 29	7.74
	5M24 HT	61	49–51	50	Av 27.5	7.21
2	5S24 AC	48	65–68	66	28 – 32	9.80
	5S24 HT	52	60–63	62	Av29.5	9.31
3	10M24 AC	50	61–65	63	24 – 27	9.74
	10M24 HT	54	59–61	60	Av 25.5	9.42
4	10S24 AC	47	68–71	69	23 – 26	10.02
	10S24 HT	50	66–68	67	Av24.5	9.84

TABLE 3

PLS DATA OF Cr–Mn IRON SAMPLES

Sl. No.	Sample	PLS Parameters									
		AC		HT		AC		HT		AC	HT
		τ_2 (ps)	τ_{av} (ps)	τ_2 (ps)	τ_{av} (ps)	I_1 (%)	I_2 (%)	I_1 (%)	I_2 (%)	k (×10^9)	k (×10^9)
1	5M24	215.3	142.2	197.2	134.8	67.5	32.5	69.2	30.8	4.52	4.22
2	5S24	220.8	145.8	212.7	141.8	65.9	34.1	67.1	32.9	4.75	4.54
3	10M24	226.2	144.3	224.0	143.0	68.6	31.4	69.5	30.5	4.24	4.14
4	10S24	222.5	143.6	215.7	140.3	68.3	31.7	69.3	30.7	4.35	4.25

Hence, for example, 5 % Mn bearing, 24 mm sized, metal mould sample is designated as 5M24. Figures 1 and 2 show the features of light micrograph in respect of the heat-treated 5M24 and 5S24 samples, respectively. The error (i.e. coefficient of variation) obtained on the measured values in respect of erosion is well within 5 % and for the hardness value, the error is ± 1HRc.

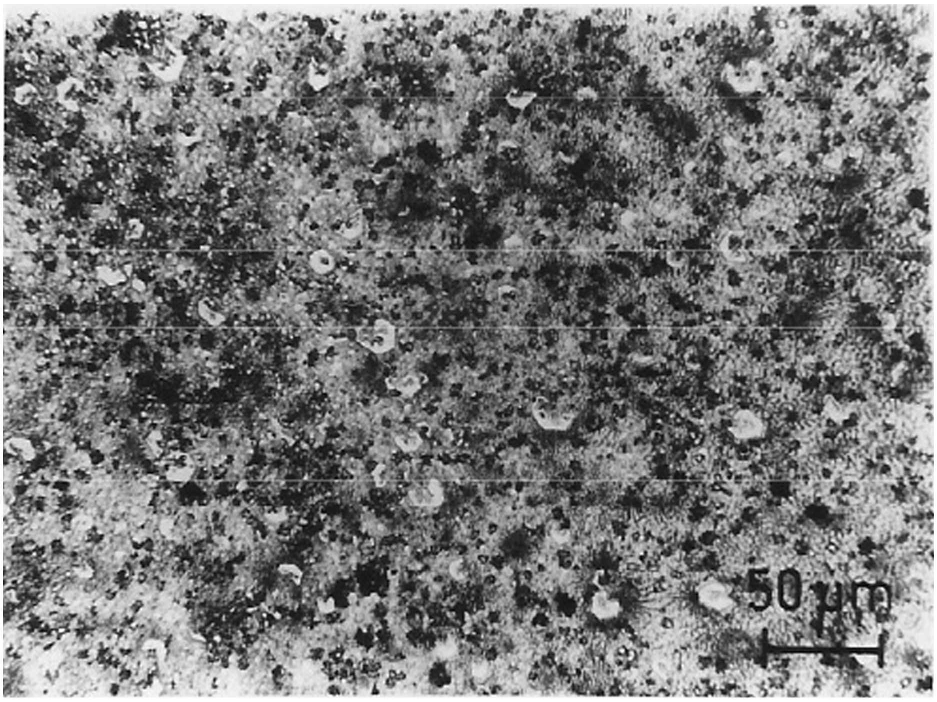

FIG. 1 MICROSTRUCTURE OF 5 % MANGANESE BEARING METAL MOULD HEAT-TREATED (24 mm) SAMPLE

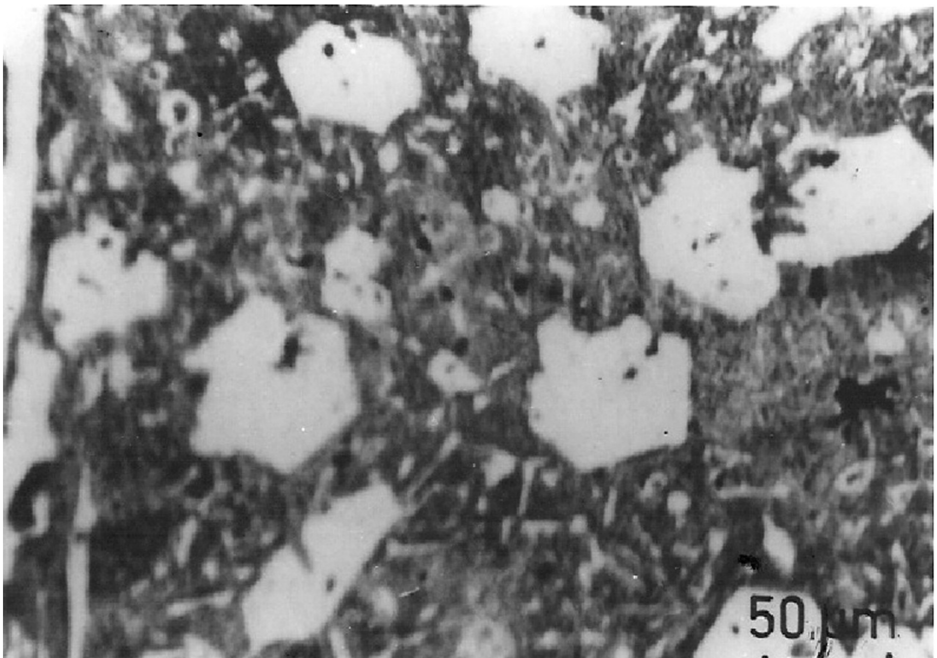

FIG. 2 MICROSTRUCTURE OF 5 % MANGANESE BEARING SAND MOULD HEAT-TREATED (24 mm) SAMPLE

3.1 Mould Type Affecting Erosion and PLS Parameters in the As-Cast Condition

From Table 2, it is seen that the erosion volume loss is higher and hardness is lower for the 5 % Mn bearing sand mould samples compared to metal cast counterparts. Similarly, for the 10 % Mn-bearing cases, the erosion loss is lower and hardness is higher for metal mould samples compared to the sand mould ones. Further, the erosion data obtained get good credence from the microscopic point of view, wherein 5S24 has shown randomly distributed medium-sized carbides plus massive carbides (marked ‘M’) with higher austenitic content (66 %; marked by an arrow) and hence exhibited lower hardness (48 HRc). On the other hand, 5M24 reveals small-sized primary carbides (marked ‘S’) with occasional hexagonal carbides (marked ‘H’) in the lower austenitic (57 %; marked by an arrow) resulting in higher hardness (55 HRc). Coming to 10M24 metal and sand cast samples, 10M24 has yielded longer primary carbides with 63 % austenite plus higher CV, thus contributing to lower hardness (50 HRc). 10S24 has exhibited primary long (large) carbides in a (the errors obtained on the measured values in respect of erosion are well within 5%) predominantly austenitic (69 %; marked by an arrow) matrix resulting in lowering the hardness (47 HRc). 5M24 and 10M24 have shown smaller-sized carbides with lower RA, thus contributing to higher hardness as well as higher erosion resistance (lower erosion loss) compared to 5S24 and 10S24, respectively.

The mean range of positrons in the Cr–Mn iron systems has been experimentally found to be 32.93 μm. As the section size of the casting is quite thick (24 mm), positrons are expected to probe the bulk of the system. It is known that the defect profile at the surface is not necessarily the same as that of the bulk; however, the defect profile of the bulk influences the surface properties. This issue has been reported from the surface studies carried out in various metals and semiconductors using slow positron beams [10].

The lifetime in these samples in the absence of defects should be in the range 99–107 ps, Further, the theoretical estimate of the expected defect lifetime for these systems assuming monovacancy type should be about 186 ps. But the second lifetime obtained in the range 197–220 ps, which is higher for these samples, indicates that there are defects in the sample. This certainly points out to the fact that the defects are not necessarily of monovacancy type but of bigger size evolved in the Cr–Mn–Fe system during the solidification process in both metal and sand moulds. Although the mean range of positrons in all these systems is the same, their defect lifetime varies with respect to the mould variety used as explained below. In the metal and sand mould employed, the heat transfer process appears to be different with the result; a transformation in the microstructure takes place as a result of varied cooling rates prevailed in the moulds. As the fraction of positrons probing the single interface is negligible due to the mean positron diffusion length of few hundred nanometers, very little trapping is expected from the interface. These facts indicate that most of the positrons are getting annihilated in the bulk state as seen from the I_2 values given in Table 3. Thus, lower positron lifetime and specific trapping rate exhibited by 5M24 indicate the fact that it has less number of defects (trapping rate) and they are of smaller sizes compared to 5S24 as seen from Table 3. Similarly, lower specific trapping rate and marginal variation in the lifetime of positron in 10M24 are indicative of lower defect concentration and decreased defect size compared to 10S24. Further, the data trends seen in PLS are in line with the trends noticed in the erosion experiments.

Hence, the heat transfer coefficient will also be higher in the metal mould than in sand mould. No attempt has been made in the present data trends, thus emphasizing the fact that the metal mould samples are having smaller-sized carbides and less defect concentrations compared to the sand mould counterpart.

The change in solidification process on account of mould variety adopted results in varied heat transfer characteristics [11]. As the solidification rate prevailed in the metal mould is higher than the sand mould in view of higher thermal conductivity (k) and lower specific heat (c) prevailed in the metal (k:52 W/m°k) mould compared to sand (k:0.325 W/m°k) mould which has lower k and higher c, the metal mould has faster cooling rate.

3.2 Effect of Mould Variety on Erosion and PLS Parameters in the Heat Treated Condition

In Table 2, the erosion and hardness data, RA % and CV % of the heat-treated samples are shown. It is noted from the above Table that 5M24 shows lower volume loss and higher hardness compared to 5S24, as 5M24 (Figure 1) shows small-sized but fine primary carbides (marked 'F') plus hexagonal carbides (marked 'X') with lower RA (50 %; marked by an arrow) content and higher hardness (61 HRc) compared to 5S24 (Figure 2), which has yielded randomly distributed bigger (marked 'B') plus medium-sized carbides (marked 'M') with RA content of 62 % (marked by an arrow) and hardness of 52 HRc. The heat-treated 10M24 sample (Figure 3) has shown medium-sized primary carbides with lamellar type of long carbides in an austenitic (60 %) matrix with hardness and CV values of 54 HRc and 25.5 %, respectively. On the other hand, 10S24 reveals multi-directional medium-sized primary carbides and lenticular (long) carbides with hardness of 50 HRc and CV of 24.5 % in an austenitic (67 %) matrix. It is reported in the literature [12,13] that better properties are achieved when the grain/particle sizes are smaller and finer in the matrix.

It is true in the case of 5M24 that the globular type of fine carbides formed during the heat treatment and toughening of the matrix (lower RA content) are the key factors responsible for improved erosion behavior in 5M24 compared to 5S24 as well as 10M24 and 10S24.

It is noticed that PLS data after the heat treatment of the samples showed a decreasing

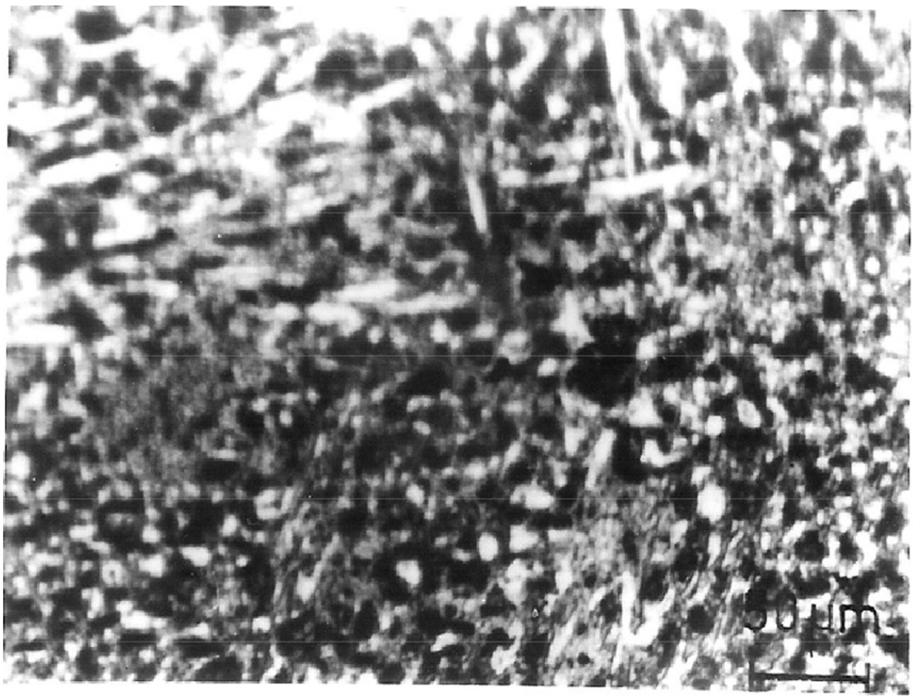

FIG. 3 MICROSTRUCTURE OF 10 % MANGANESE BEARING METAL MOULD HEAT-TREATED (24 mm) SAMPLE

trend compared to the as-cast samples. This is attributed to annealing out of some defects and also resulting in smaller defect sizes and this correlates well with the globular type of carbides in respect of the heat-treated sample 5M24. The data in Table 3 further suggests that size and concentration of defects in the bulk showed some decrease in the metal mould sample compared to sand mould ones.

In summary, it is stated that higher erosion loss and higher positron trapping rate (more defects) observed in the as-cast and heat-treated samples of 5S24 and 10S24 may be attributed to longer time available for the diffusion of molten material and the evolution of bigger size carbides in the sand mould samples and vice versa is true for metal mould samples. In other words, the globular type of carbides were formed in faster cooling conditions prevailing in metal mould casting, especially in the heat-treated condition due to the spheroidization of carbides.

3.3 Surface Defect Characterization using the Slow Positron Beam (DBAR)

The variation in *S*-parameter as a function of incident positron energy and the mean implantation depth of the positron beam is shown in Figure 4. The mean implantation depth of mono-energetic positron beam having incident energy *E* in a material with density ρ (gm/cc) is determined using the following equation

$$\langle Z \rangle = \frac{40}{\tilde{n}} E^{1.6} \tag{3}$$

where, (Z) is expressed in nm and *E* in keV.

The *S*-parameter profile of 5M24 is seen to be different from the other two samples, namely 10M24 (AC) and 10M24 (HT). The surface as well as the bulk values of the *S*-parameter in 5M24 are seen to be lower than that of 10M24 (AC) and 10M24 (HT) samples according to the data shown in Table 4. It indicates that the defect concentration increases with the increase in Mn percentage at the surface. The diffusion length in 5M24 is also higher compared to 10M24 samples, suggesting a low concentration of defects in 5M24. However, the profiles of *S*-parameter among the two 10M24 (AC and HT) samples are almost identical, indicating near absence of any modification of defect structure near the surface following heat treatment.

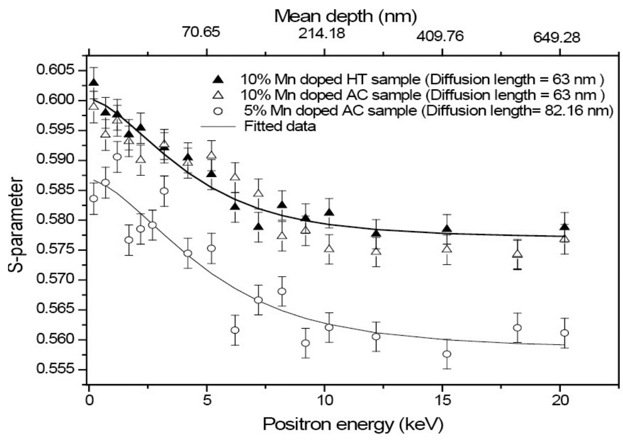

FIG. 4 S-PARAMETER VS POSITRON ENERGY IN DBAR EXPERIMENT

TABLE 4			
VEPFIT FITTED PARAMETERS FROM DBAR EXPERIMENTS			
Sample Identification	**S-parameter bulk**	**S-parameter surface**	**Diffusion length (nm)**
5M24 (AC)	0.5585	0.5885	82.16
10M24 (AC)	0.5730	0.5960	63.51
10M24 (HT)	0.5769	0.6020	63.51

4.0 CONCLUSION

- The following conclusions are drawn from this work. The 5 % and 10 % Mn-bearing metal mould samples, irrespective of the metal mould samples, show a lower defect concentration irrespective of the Mn content compared to sand cast counterparts, as the former one exhibits lower positron lifetime, reduced intensity and higher specific trapping rate.
- The heat treatment has brought about microstructural transformations involving change in the carbide morphology

(spherodization of carbides) and toughening the matrix by way of reduction in austenite content in 5M24 which has resulted in increased hardness and higher erosion resistance compared to the heat-treated 10M24 as well as its as-cast counterpart.

- The defect sizes (lifetime) and their defect concentration decrease in 5M24 following heat treatment compared to its as-cast sample as well as 10M24 as-cast and heat-treated samples.
- S-parameters from DBAR experiments show lower values, thus indicating lower concentration of defects in 5 % Mn-bearing sample compared to 10 % Mn-bearing as-cast and heat-treated samples.
- A good correlation has been unambiguously established among the propertiesm, viz. hardness, erosion, DBAR and SPLS parameters.

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