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Comparison of Cheap Imported Stainless Steel Samples with Indian-made Samples and a Crystalline Phase Based Methodology for Bench-marking them

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In the emerging South Asian markets, from the commercial as well as engineering point of view, performance and quality of any local engineering product like steel structures vis a vis similar but imported, cheap materials is always an issue. In this paper, as a representative case, we discuss the composition, crystalline phase and microstructure of two most common stainless steel grades manufactured locally by the major government of India sponsored (GOIS) Steel maker and compare them with similar cheap imported items that some local private businesses prefer due to slightly lower costs but having similar materials usage parameters. We have also used wet etch based micrographic analysis to compare surface morphology and have also done composition analysis in our results. It is shown that even if ASTM standards of composition are followed, since their crystalline phase components are not the same, their performance and properties are never comparable. More stringent quality bench-marking of such alloy materials for checking repeatibility of batches and processes using X-ray diffraction and Raman spectral data analysis is thereby proposed.

Keywords: Alloys; Stainless steel; Benchmarking procedures

1 Introduction

In today's modern world, industrial progress of any segment in any country is determined by the number of polymers-plastics, the number of different metal alloys and the total number of medical active ingredients it can independently produce. In the emerging South Asian markets, the performance and quality of any locally made engineering product in any segment from polymers-plastics to different metal alloys is judged vis a vis similar but other imported, cheap materials from other Asian countries. Among steels, special steels and stainless steels have a niche area of application on routine basis in different segments of our modern society in medical, domestic and industrial segments. Stainless steels have different grades and are generally considered to be defined by the American Iron and Steel Institute (AISI) and Society of Automotive Engineers (SAE)^{1, 2}.

An alloy is a mixture of different known but controlled amounts of elemental constituents in certain proportions. Steel may be defined as a primarily iron matrix based alloy containing carbon and other alloying additions with controlled constituents, composition and also with a controlled and known microstructure and having a known experimental repeatibility³. As the name suggests, stainless steel is a type of steel i.e. it is an iron matrix which is less susceptible to stain or scratch marks or corrosion from even quite harsh ambient corrosive conditions during its normal course of usage, having less than 1% of carbon in its constituent. It has certain crystalline phases and certain amorphous phases of known composition giving its the required mechanical strength, and having silicon, nitrogen, sulfur, aluminium, titanium, nickel, chromium, copper, selenium, niobium, and molybdenum as the other constituents in such relative proportions in the crystalline or amorphous form that all pitting and other forms of corrosion are negligible even with time due to its self passivizing upper coating formation leading to a chemical stability^{1,4}. In this context, here, а representative case, we discuss as the microstructure, crystalline phases and composition of two of the most common stainless steel grades manufactured locally by a major government of India sponsored (GOIS) steel maker and compare them with similar cheap imported items that some local private businesses prefer having similar materials usage parameters, due to slightly lower costs and dumping issues. It is not possible to give a scientific

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comparison for each and every imported Stainless steel product or other imported steel seen in Indian market but some representative cases like these can be considered to convey certain issues in general. We have tried to make out a scientific case for more enhanced usage of locally made metal alloys, their better homogeneity, considering better crystallinity of certain elemental phases and amorphousness of other alloy or elemental phases, elemental composition, composition stability w.r.t. known composition tables as may be seen from alloy data sheets¹, crystalline and associated better performance issues. As of today, from this bench mark, alloys are currently designated by their elemental composition alone, and not by which crystalline or amorphous phases are present or absent. Samples are then tested in terms of their yield strength, elasticity, ductility, hardness, corrosive resistance, non-magnetic performance. microstructure and such other mechanical properties. Instead, we are trying to propose judging such alloy samples not just based on their composition alone, but also on their extent of different crystalline and amorphous phases, average grain sizes - properties that are much more fundamental and less time consuming to measure and characterize on a mass scale during industrial processing. In this proposed methodology, using routine X-ray diffraction patterns (XRD) and Raman spectra at room temperature, it should thus be possible to segregate different alloy samples of proper crystalline and a few amorphous phases of interest having the same elemental composition as against those samples that do not have all such crystalline phases of appropriate proportions. It will lead to better value for money to the citizen on the street and better quality of downstream products made with these with better durability in the local market and these will possibly be more agreeable for the export market too.

Stainless steel, in general, has the reputation for lack of corrosion under normal circumstances. Its' utensils do not corrode or crack easily^{4.} In the last few years, it has been observed that a lot of non- GOISs made stainless steel products are being sold in the Indian open market. After a while, however, most of these items were seen to corrode or crack during routine domestic usage very quickly as observed by many Indian citizens, including the authors themselves, a phenomena not seen or heard of, even a decade or two earlier, when GOIS SS materials were dominating in the Indian market. The vendors of these items informally declared that these were made from imported steel sheets from other Asian countries^{5, 6}. So it was felt necessary to investigate the scientific issues behind these failures.

We have used experimental data from X-ray diffraction, Raman spectral data, used different chemical wet etchants like Nital and Glycergia respectively and then observed the samples' using electron microscopy (SEM) images, and experimental data from X-Ray fluorescence (XRF), Electron microscopy related electron diffraction based x-ray data analysis (EDAX) and spark plasma based optical emission spectroscopy data to base our observations. Our objective is to understand the phenomena and also educate any common Indian reader on why local stainless steel products should be preferred from the quality or engineering point of view. Such SS quality is also associated with any downstream secondary or tertiary SS based product people wish to manufacture leading to such locally made but better quality stainless steel based products. These are discussed below.

2 Experimental

(b) X-Ray Fluorescence (XRF) spectra related data collection and analysis was done on these samples to know their nominal concentration and composition of the alloys. A Rigaku ZSX Primus wavelength dispersive X-ray fluorescence spectrometer (XRF) was used for checking the composition of these metal SS samples. This spectrometer has an Rh-target endwindow sealed X-ray tube, operated at 4 kW as excitation source and a scintillation counter as the detector. It uses an X-ray Ka spectral lines for elemental analysis at a tube rating of 50 keV and 60 mA and a LiF(200) based analyzer crystal. These data was subsequently correlated with data from EDAX. The data was also correlated with elemental analysis data collected using Spark Plasma based optical emission spectrometry (SP-OES), using an Angstrom V-950 SP-OES, USA.

Etching of these alloy samples were done using a modified nital solution, comprising 100ml of 2-Propanol and 10 ml of Nitric Acid in as is condition. All chemicals were sourced from Rankem-India. These samples were etched for up to 20 minutes in each case and thereafter the reaction was stopped by decanting and flooding. 2-Propanol was chosen for convenience as it is in better compliance with local laws. It was however seen that in spite of etching for such a long time duration, for most of the samples, the luster did not significantly fade. Choice and justification for the etchant used i.e. modified nital was based on literature reports⁷. Other suggested etchants were then considered like Glycergia, consisting of 15 cc of H-Cl, 10 cc of glycerol, 5cc HNO₃, taken as is and used at room temperature for 5 minutes. It was seen that after this time, the shiny nature of all the sample surfaces disappeared and it was no longer reflecting. These choices are based on past published data on the success of revealing the surface defects of such smooth polished metal surfaces⁸. Morphology and elemental analysis studies were done through energy dispersive x-ray spectroscopy (EDAX) using a ZEISS EVO MA10 SEM with EDS: OXFORD INCA 250 ISIS system. All samples were inter-compared at a magnification of 5000X.

The X-ray diffraction (XRD) analysis of our samples was done using a Philips XpertProPlus Powder X-ray diffractometer having a copper target. It was operated at 40 kV and 40 mA. In this case, calibration of the instrument was done using a Silicon electronic grade wafer before taking the data. Based on obtained XRF and EDAX data, the nominal elemental content was deduced and it was possible to understand all possible crystalline phases that can be expected in each case during an XRD analysis. Those that were not in this list were not considered for possible phase analysis for that specific sample. It may be noted that the identification of all phase compositions in totality is crucial for a better understanding of the likely performance limitations or enhancements and degradation processes that may be subsequently observed.

Raman spectra of our samples were recorded using a Renishaw inVia Raman Spectrometer, UK having a

laser excitation source emitting at 514 nm, with exposure times of up to 120 or 180 seconds. Such higher times were at times needed to get identifiable features. Based on XRD data analysis, in each sample, the crystalline phases present were identified. Based on such data, Raman spectral peaks were identified through a phase correlation and associated literature reports on Raman spectral lines of such crystalline phases as discussed below. Detailed background subtraction was done using Origin 7.5 software and the data was obtained in a presentable format.

3 Results and Discussions

Table 1 and Table 2 shows composition profile of the four representative samples of stainless steel (SS). The first sample, designated as (a) corresponds to GOIS made SS-430. The second sample, designated as (b) corresponds to GOIS made SS-304. The third sample designated as (c) and the fourth sample, designated as (d) are representative samples sourced from the Indian market but which are made from cheap imported SS made in other Asian countries.

As per EDAX data, other than Fe, C, O, Cr (a,b,c,d), Fe (a,b,c,d), Ni (b,d) and Si (c,d) are the major constituents in general most of the time. However, in the lower atomic numbers (z values), EDAX is not known to be very accurate in quantification. XRF could not detect oxygen in any of the samples. In the case of stainless steel, presence of oxygen is known to be quite unlikely as per ASTM data ^{1, 2}. This only reinforces the usually held belief that at smaller Z values, EDAX is not very accurate or sensitive. As an extra, Mn (b,c,d) and Mo (b) could also be detected by XRF in some of the samples. But both techniques largely detected Si in the cheap imported samples (c,d). XRF also detected Ni only in (b,d). Even though the obtained numbers are given in

Tab	ole 1 — Showi	ng EDAX da	ta of different	t Stainless stee	l samples and	their composi	tion.	
Stainless Steel Samples	C (wt %) O (w		%) Cr (wt %)		Fe (wt %)		wt %)	Si (wt %)
M-NK-1 (a), SS-430	1.63	1.	56	14.65	82.16	0		0
M-NK-2 (b), SS-304	1.68	2.	07	16.25	69.37	10	.63	0
M-VJ-1 (c)	2.64	2.	65	10.21	83.71		0	0.78
M-VJ-2 (d)	2.99	3.33		10.49	75.31	6	5.9	0.98
Ta Stainless Steel Samples	C (wt %)	O (wt %)	Cr (wt %)	Stainless steel Fe (wt %)	samples and th Ni (wt %)	Si (wt %)	10n. Mn (wt %)	Mo (wt%)
1	C (wt %) 30.27	O (wt %)	Cr (wt %) 12.27	Fe (wt %) 57.45		S1 (wt %)	Mn (wt %)	Mo (wt%) 0
M-NK-1(a), SS-430	00121				0	0	0	•
M-NK-2(b), SS-304	0	*	19.41	69.07	9.4	0	1.85	0.26
M-VJ-1(c)	12.13	*	12.50	66.03	0	0.48	8.86	0
M-VJ-2(d)	8.75	*	14.39	65.71	3.77	0	7.37	0
* Could not be detected.								

the table, due to lack of a standard sample, their veracity is not being discussed in detail here, as evidently the impractically high carbon content numbers obtained suggests that the XRF quantification without a standard SS sample may not be wise in this case at the moment.

SP-OES data for three samples GOIS SS-430, GOIS SS-304, and one of the SS imported samples sourced from the market SS-VJ-1 are now discussed. This methodology is considered more accurate from the quantification point of view. Sample SS-VJ-2 could not be analyzed by this method as its width was too small for minimum sample size requirements by this experimental technique on this particular instrument. In this method of analysis, GOIS SS-430 had 80.73% Fe, 0.06 % C, 0.17% Si, 0.28% Mn, 0.05% P, 0.03%S, 0.2% Ni, 18% Cr, 0.11% Mo, 0.07 %Cu, 0.05% V, 0.02% Al, 0.01% Ti, 0.02%Nb, traces of B, 0.02%N, 0.04%W, traces of Zr, 0.05% Co, traces of Pb, 0.04% Sn. GOIS SS-304 showed presence of 69.4% Fe, 0.04%C, 0.2%Si, 1.6%Mn, 0.04%P, 0.02%S, 8.7%Ni, 19.3%Cr, 0.18%Mo, 0.1%Cu, traces of Ti, Nb, 0.03%V, 0.02%N, 0.16% Co, 0.04%W, traces of Sn, traces of B, 0.01% Al. SS sample M-VJ-1 showed presence of 73.5% Fe, 0.1%C, 0.4%Si, 10.1%Mn, 0.05% P, traces of S, 0.4% Ni, 14.5% Cr, 0.04% Mo, 0.6% Cu, 0.03%Co, 0.01% Al, 0.01% Ti, 0.01% W, 0.07% V, traces of Nb, traces of Sn, and 0.2% N. So it is seen that cheap imported SS samples like SS-VJ-1 are also not compatible chemically with the typical GOIS SS-430 or SS-304 that they were expected to mimic.

Figure 1 shows the X-ray diffraction (XRD) related graphs of the four different representative samples of stainless steel. At the first glance itself, looking at all the possible crystalline phases that were possible from composition of the elemental constituents as above. the biggest perceptible difference in the different representative SS samples being studied here is that the different crystalline phases present in each of the samples in each of the cases and their relative amounts are different. The other constituents possible are all in the form amorphous phases in each of these four cases. Sample (a) of GOIS SS-430 has two sharp XRD (2Theta) peaks at 44.77° and 65.12° respectively. As per JCPDS cards (06-0696), both of them correspond to sharp crystalline Fe peaks. Sample (b) of GOIS SS-304 has three XRD 2Theta peaks at 43.73°, 50.95°, and 74.68° respectively. As



Fig. 1 — The XRD patterns for different stainless steel (SS) samples available in our local market - (a) GOIS made SS-430, (b) GOIS made SS-304, (c) and (d) are from two other cheaper SS samples produced in other Asian countries and available freely in the local Indian market.

per JCPDS card 33-0945, Ni-Cr-Fe phases has peaks in these positions. As per JCPDS card 18-0646, Fe-Ni has peak positions at these places. As per JCPDS card 38-0419, Fe-Ni phase also has peaks in these positions. So, it may be said that that this GOIS SS sample SS-304 (b) is a combination of several phases of its constituents based on the preparation and temperature cycling processing used. As there was no trace of the crystalline Fe, it may be said with a certain degree of certainty that Fe exists in elemental form in this sample (b) only as amorphous or nanocrystalline form. That will be known and confirmed after post chemical etch analysis of these SS samples as discussed in the subsequent paragraphs.

A literature survey in the area of stainless steel SS-430 and SS-304, made in other parts of the world deserves to be discussed in this context. Armouche et al.⁹ have reported that XRD peaks for their SS-430 samples were at around 44°, 65°, 84° respectively. Carvalho et al.¹⁰ have reported that XRD peaks for their SS-430 samples had small peaks at around 10° , 28° , 38° , 51° moderate peaks at around 35° , 43° , and a strong peak at 55°. Mahato et al.¹¹ have also reported that their SS-450 samples as having XRD 2Theta peaks at 38°, 45° and 66° respectively. Buhagir et al.¹² have reported similar SS-430 samplers as having XRD peaks at 44° and 50° respectively. So it may be seen that even in different SS-430 samples reported from different parts of the world, their crystalline and amorphous phases are different from the GOIS SS-430, even though their elemental chemical composition are quite similar.

Ozturk and Williamson¹³ have discussed SS-304 where their XRD peaks are at around 42° , 46° , and 50° or so. These peaks are markedly different than that seen in our GOIS SS-304 [Fig. 1 as discussed above]. De et al.¹⁴ discussed a variant of this Stainless steel which has XRD peaks at around 42° , 44° , 47° , 51° , 62° , 65° , 75° , 82° , 90° and with a grain size of 8 microns. These peaks profiles also do not match with our GOIS peak profile. Moser et al. 15 have reported XRD peaks of their SS-304 at around 45°, 51°, 65°, 75° , 83° and 90° , while their relative peak intensities varies with different temperature treatments. But these do not match with the peak profiles of our GOIS SS samples. Wang and Sun¹⁶ have also discussed XRD data for their SS samples. Their samples show XRD peaks at around 44°, 51°, 75° respectively. These data almost match with the peaks of our SS samples. Singh et al.¹⁷ have observed XRD peaks in their SS-304 samples at around 44° , 64° , 82° respectively. These also do not exactly match with our samples' data. Oing *et al.*¹⁸ have also published their SS-304 XRD data where the peaks are reported to be at around 43° , 51° , 75° , 90° respectively. These almost match with our data. So, all of these representative data taken from other scientific workers published results suggest that all these different groups have different formats of thermal treatment protocols for their SS-304 sample preparation leading to different crystalline formats, even though their samples' chemical composition constituent profiles may be quite similar as per ASTM data composition and constituent requirements. So it may be said with a reasonable degree of certainty that their performance properties will also be quite different considering the large differences in their crystalline structures based on the manufacturer.

XRD peaks of the first representative cheap imported SS sample as in Fig. 1(c) show XRD peaks at 43.46°, 50.43°, and 74.42° respectively. XRD peaks of the second representative cheap imported SS samples as in Fig. 1(d) show XRD peaks at 43.47°, 50.53°, and 74.42° respectively. Their relative differences from the crystalline point of view is the difference in relative intensity of the peaks and also in changes in their full width at half maximum (FWHM) values. There are also other differences in terms of relative chemical composition and wet etch performance, as has been discussed in subsequent paragraphs below.

So it may be seen that in general, some SS samples may be cheaper in price, and in spite of similar elemental chemical composition, due to differences in crystalline structure, their performance properties may vary appreciably as it is finally the crystalline properties, micro-structure and average crystalline size, the associated amorphous phases and their relative contents-amounts that finally determine their performance properties and their relative improvements or their relative deterioration. So we now have a possible scientific explanation on possibly why some steel products may perform better, even though they cost a little more.

Figure 2 shows the background subtracted Raman spectra of these four representative SS alloy samples. Fig. 2(a) corresponds to Raman spectral plotted data for sample M-NK-1 i.e. GOIS SS-430. Fig. 2(b) corresponds to data for sample M-NK-2. GOIS SS-304



Fig. 2 — The Raman spectral pattern for (a) GOIS made SS-430, (b) GOIS made SS-304, while (c) and (d) are from two other cheaper SS samples produced in other Asian countries and available freely in the local Indian market.

304. Fig. 2 (c) & (d) are for the two samples sourced from the market which were made from cheap imported SS sheats from other Asian countries - their supposed intended uses are supposed to mimic those for SS-304 type samples. It may be seen that broadly speaking, the overall Raman spectra does not match in any two of these over the whole range of measurement. That should be expected due to the difference in their alloving content and variation in crystalline structure and amorphous phase content as a result. Fig. 2(a) for GOIS SS-430 shows a small peak at 315 cm⁻¹, a relatively broad peak at 758 cm⁻¹ (with no separate discernible - distinct peak at 840 cm⁻¹), at 1406 cm⁻¹ and another one at 1624cm⁻¹ respectively. Fig. 2(b) for GOIS SS-304 shows a joined peak at 772cm⁻¹ and 843 cm⁻¹, another joined peaks at 1360 cm⁻¹, 1446 cm⁻¹ and another peak at 1626 cm⁻¹ respectively.

On the other hand, in the case of the two representative imported SS samples, Fig. 2(c) has a peak at 233 cm⁻¹, at 685 cm⁻¹, and two joined peaks at 1395 cm⁻¹ and 1620 cm⁻¹, while Fig. 2(d) has a peak at 290 cm⁻¹, 764c m⁻¹, 1404 cm⁻¹ and 1627 cm⁻¹ respectively. There is significant variation in the spectral profile of Fig. 2(c) and Fig. 2(d), even though their XRD profiles were quite similar. This also suggests that their internal microstructure and alloying structure is sufficiently different. EDAX/XRF data also suggests so.

In each of the above cases, the Raman peaks at around 760cm⁻¹ may be attributed to Fe-Cr, Fe-O to the extent they are present in crystalline form, at times in small quantities in these SS samples¹⁹. The Raman peak at 840 cm⁻¹ is attributable to Si-C present in small amounts^{20,21}. The Raman peak at around 1400cm⁻¹ is attributable to traces of Fe-C impurities in crystalline form²², while the Raman peak at 1620 cm^{-1} is also attributable to other vibration modes due to traces of Fe-Cr present in crystalline form^{22,23} respectively. The noisy signal is due to the low content of the material in the matrix. Raman peaks at 1360 cm⁻¹ are attributable to crystalline Fe-C²². But it is a bit shifted, possibly from internal stress, while those at 1446 cm⁻¹ are reported to be due to Fe, Cr-Fe, but shifted the other way. Peaks at low wave numbers for such SS samples, including in the range 330 cm⁻¹ 700-800 cm⁻¹ are ascribed to be due to Ni-Fe, Cr-Fe²⁴ in general.

Figure 3 shows the different wet etched SS samples when etched using Nital etching. Nital was used here



Fig. 3 — The Nital etched Stainless steel samples: (a) corresponds to GOIS SS430, (b) GOIS SS304, (c) and (d) correspond to two representative examples of etched SS samples sourced from market which used imported sheets from other Asian countries.

initially as a test case, as we were initially also observing its etching performance on several other different non-SS grades of steel, which we have reported on separately²⁵.

As in other cases, Fig. 3(a) here represents GOIS SS-430, Fig. 3(b) here represents GOIS SS-304 grade representative sample in etched condition. Figs. 3(c) & (d) again represents two representative cheap imported SS of similar grade from other Asian countries. For comparison, their magnification has been kept the same in each case (5K X). The samples had been kept dipped in nital for 20 minutes in each case, at room temperature, in the hope that the sample shine would be diminished but in vain. In case of GOIS SS-430, no grain boundary could be detected [Fig. 3(a), though, traces of sample polishing and possibly hot rolling related pressing effects could be detected. In the case of GOIS SS-304, a lot of grain

boundaries could be detected, with average grain sizes in the range of 6 to 10 microns [Fig. (3b)]. In the case of Fig. 3(c), the contrast was too poor to see any worthwhile grain boundaries. In Fig. 3(d), the sample surface showed traces of all most cold worked pressing related streaks.

All these four SS samples were then treated separately with Glycergia as wet chemical etchant for 5 minutes each at room temperature and their surfaces were again observed under the SEM under similar magnification conditions. In this case too, the sample nomenclature remained the same. Fig. 4 shows the corresponding relevant images for these representations. It was seen that, this time, the etch performance was much better, the sample luster had disappeared in each case and all four sample surfaces showed crystalline artifacts. All four samples had bubble type features of some gas bubble or blow-hole



Fig. 4 — The Glycergia etched Stainless steel samples: (a) corresponds to GOIS SS430, (b) GOIS SS304, (c) and (d) correspond to two representative examples of etched SS samples sourced from market which used imported sheets from other Asian countries.

residue ²⁶, possibly during the sample preparation stage. Fig. 4(a) also showed linear streaks possibly due to sample sheet preparation related semi hot rolling requirements. Fig. 4(b) also had such linear streaks from sample sheet preparation related semihot rolling requirements. In addition, it also showed distinct grain boundaries of the sizes of about 10-15 micron or so. Fig. 4(c) showed quite a few such gas bubbles and etch pits, in addition to the linear streaks from sample sheet preparation related rolling requirements. Fig. 4(d) also showed linear streaks from rolling requirements during sheet preparation, along with the gas bubble residues. Due to the difference in materials content and their different crystalline and amorphous phases, the image background of the micrographs were also different.

So it is seen that the representative GOIS and cheap imported SS samples are quite a bit different in

every aspect even from the SEM micrographic point of view.

4 Conclusions

It has been shown using XRD data of four different stainless steel (SS) samples, two sourced from local GOIS and two others as representative cheap Asian imports that on comparing them with each other and also comparing with published literature based data of similar stainless steel samples that even though in the same SS grade, their elemental compositions may be similar, their crystalline features like extent of crystalline phases and extent of amorphous phases may be quite different, making their respective materials' performances quite different and distinct. So often, the cheaper imported SS samples may corrode, while the GOIS SS samples may not in that time period. It also reinforces the observations seen using SS utensils made from GOIS on their good long term performance as compared to samples using cheap imported SS sheets. Elemental composition of these SS samples was checked using XRF, EDAX and SP-OES. Raman spectral data independently reinforced the XRD crystalline phase related data seen here. Wet etch micrographs of these samples were also seen to indirectly show their composition, crystalline and amorphous phase differences.

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Data Availability Issue

Raw data from the experiments at our end are in principle available upon reasonable request and reasonable cause from the corresponding author.

Financial Interests

None of the authors have any direct financial interest in SAIL or in commercial large scale steel making as on date. We also have no conflict of interest with any Indian or overseas steel maker as on date.

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