THE DESIGN OF X80 TO X120 STEELS WITH INCREASED CARBONIC ACID CORROSION RESISTANCE

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ABSTRACT

A methodology for increasing the CO₂ corrosion resistance of microalloyed steels containing at least 3% Cr has been described previously with the intention of achieving strength levels in the range 552 to 828MPa (80 to 120ksi). The current work reviews a further development of this class of steels in which the role of Si, Cu, Ni and V are investigated. Whilst the study has confirmed that Si, Cu and Ni can be used to raise strength the more interesting results concern the role of V in improving corrosion resistance in carbonic acid. Small scale corrosion tests showed that the corrosion behaviour both in long term tests in carbonic acid solutions, pH 5.4, and in linear polarisation resistance (LPR) tests at pH 5.4 and 8.4 were influenced by microstructure and the metallurgical state of the V microalloying addition. This study showed that V addition was generally beneficial in improving the corrosion resistance of 3%Cr steels but particularly so when the V was in the form of very small coherent precipitates or retained in solution at the end of processing. The advantage conferred by V was also noted in larger scale melts (up to 50kg) and these results suggested that the corrosion resistance could be further optimised by processing. More significantly, these benefits were consistent with earlier laboratory and limited field testing of commercial scale heats but the goal of developing an enhanced corrosion resistant steel capable of consistently achieving X100 (690MPa) may only be realised by careful attention to processing. Some proposals for further optimising this novel approach to steel design are outlined.

KEYWORDS

High strength steels, X80, Corrosion rate, Carbonic acid, Alloying, Vanadium.

INTRODUCTION

The need to improve the resistance of steels to carbonic acid corrosion has prompted both development of improved steels and an understanding of the fundamental processes involved. A particular problem arises through the type of corrosion known as 'mesa-attack' where a localised region of corrosion begins and rapidly causes perforation leaving large regions exposed to the corrosive media comparatively unaffected. The prevalence of such corrosion processes in carbonic acid media has led to the use of expensive alloyed Cr based stainless steels. Whilst eminently satisfactory for the application, usually 'down-hole', the cost of these steels is often prohibitive compared to the gain in service life. In contrast, the use of carbon steels is economic particularly when regular replacement of components, as a result of wear perhaps, is necessary. The use of corrosion inhibitors, some containing V as a vandadate compound, also prolongs life and results in cost-effective solutions to corrosion situations where alloyed Cr stainless steels may once have been the only alternative. Simultaneous with the trend of using carbon steels with inhibitors has been a growing demand for greater strength hence allowing weight and cost savings. A recently completed

international collaborative project [1] set a target of improving the corrosion performance of commonly used plain C steels by a factor of 5 at a cost penalty of not more than 1.5 times the alloying costs of the steel. Such a goal ruled out the expensive Cr stainless steels and instead emphasised the development of corrosion resistance at much lower levels of Cr addition. This work was based on the improved understanding of how Cr affects the passive iron carbonate films responsible for improving corrosion particularly in carbonic acid [2-4].

Part of this approach stressed the important role of uncombined or 'free' Cr in the surface layers in relation to the formation of protective Fe carbonate layers. Therefore a vital aspect in designing steels in this way was to ensure that other carbides, such as TiC, VC or NbC form preferentially during processing to the extent that harmful Cr carbides do not form and the 'free' Cr content is maximised. The compositions of steels based on these concepts [1] successfully demonstrated that alloying with a Cr content around 3% together with additions of Si, Mo, Cu and Ti or V could achieve the desired cost and corrosion improvement targets [5,6]. By utilising microalloying additions to the steel composition, the mechanical properties can also be modified to meet requirements by careful control of precipitation strengthening by either controlled cooling after rolling or by quenching and tempering. However, the precise role of the microalloying additions, whilst relatively well understood from the strengthening point of view, in modifying corrosion behaviour was unclear. As part of the development process for this novel class of steels a study is reported here which attempts to define the role of V in the corrosion process and the extent to which the other alloying additions, Si, Cu and Ni influence the mechanical properties. Vanadium was chosen for special attention not only because the earlier collaborative study [1,5,6] had identified that corrosion rates were reduced by adding amounts in excess of stoichiometry, but also that V had been used in commercially available corrosion inhibition treatment of carbon steels. The latter factor indicated that V as the vanadate interfered with the corrosion process and suggested that V may also be beneficial if present in an appropriate form in the steel [7.8].

EXPERIMENTAL PROCEDURES

Steel composition

A list of the steels examined in this study is given in table 1, there are 2 groups, that used for mechanical property assessment comprised 50kg experimental melts and a further group used for small scale exploration of microstructure on corrosion behaviour. The latter group were 800 gramme vacuum melts. The compositions in the first group, J3, J3Mo, J3Cu & J3CuNi, were chosen to assess further refinements to the mechanical and corrosion properties of the steel, shown as J3prev in table 1, developed in the collaborative project which had shown dramatic improvements in corrosion behaviour. The Si content, in particular, was adjusted to limit potential problems with segregation and castability compared to the J3 composition used previously. The A1 and N contents reflected commercial practise. Although the intention was to examine the role of Cu as well as V on both corrosion and mechanical properties, it was known that hot shortness due to the Cu addition would prove a problem in terms of rolling. Whilst some mechanical test data was obtained, the test programme was more limited and incomplete due to the presence of rolling defects in the rolled plate. An additional melt containing Ni was also produced primarily to reduce the tendency for hot shortness, it was considered that Ni would not be detrimental to either corrosion behaviour or mechanical properties.

	С	Mn	Si	S	Р	Al	V	Ν	Мо	Cu	Cr
50kg melts											
J3prev	0.067	0.54	0.60	<.005	<.005	.035	0.36	n/a	.31	.26	2.95
J3	0.072	0.52	0.31	<.005	<.005	.038	0.44	.011	<.005	.02	2.81
J3Mo	0.072	0.52	0.31	<.005	<.005	.034	0.43	.010	.23	.02	2.82
J3Cu	0.073	0.50	0.31	<.005	<.010	.039	0.46	.011	<.02	.50	2.81
J3CuNi*	0.075	0.50	0.30	<.005	<.010	.035	0.45	.009	<.02	.50	2.80
800g Melts											
A #	0.02	1.25	0.31	<.002	<.002	.01	<.005	<.005	<.005	.02	2.91
C #	0.03	1.28	0.31	<.002	<.002	.008	0.24	<.005	<.005	.02	2.96
VR#	< 0.01	1.05	0.32	<.002	<.002	.024	0.67	<.005	<.005	.02	2.96

Table 1 Composition of experimental steels.

* 0.35%Ni added to reduce hot shortness

Spectroscopically pure Fe was used as a base, S & P quoted as less than 0.002%.

The second group of steels, devised primarily to assess the effect of V on corrosion behaviour, A, C & VR, were based on variants of the microalloyed steel composition developed in an early phase of the collaborative project. The C and Mn contents of these steels differ from those of later phases, J3prev and the present J3 series, because of the need to avoid any risk of segregation as one of the steels in this earlier phase of the collaborative project has shown evidence of transformation from the delta-ferrite phase during processing. Aluminium was used in this group to ensure that all of the residual N content introduced during melting, ~0.002%, was present as AlN to avoid interference from precipitation of VN during heat treatment. The VR cast was chosen to reflect the condition where only V is retained in solid solution in the absence of any carbide phases.

Rolling and heat treatment

The 50kg ingots were cast as 110mm square sections and rolled to 15mm plate; rolling details are given below in table 2.

Pass Number	Thickness (mm)	%Reduction	Plate Temperature		
			K (°C)		
0	95	13.6	1519 (1246)		
1	80	15.7			
2	70	12.5			
3	60	14.3			
4	50	16.7	1476 (1203)		
5	42	16.0			
6	35	16.7			
7	28	20.0	1429 (1156)		
8	23	17.8			
9	19	17.4			
10	15	21.1	1368 (1095)		

Table 2 Rolling schedule.

Following rolling, plates were heat treated at 1213K (950°C) for 1 hour then quenched into agitated water at Swinden Technology Centre, Corus Group plc. Rotherham, UK. No measurements of cooling rate were made but the quench unit was designed to simulate normal commercial quenching practice for 25mm plate. After quenching, plates were tempered at 948K (675°C) for 15mins but heated and cooled at a rate simulating 25mm plate. Some additional mechanical tests were carried out on plate tempered at 898K (625°C).

The 800 gramme melts were cast as 50mm rod and subsequently forged to bar approximately 10mm in diameter. The bar samples were sealed into evacuated silica capsules and homogenised at 1300K (1027°C) for 3 days. These bars were then heat treated to obtain samples for corrosion testing using the following conditions:

Quenched from 1300K (1027°C), cooling rate ~5K/sec, Q1300 Quenched from 1173K, cooling rate as above, Q1173 and Furnace cooled from 1173K at a rate of 15K/min, FC1173

The objective of these heat treatments was to alter the metallurgical condition of the V. Treatment, Q1300, was intended to retain all of the V in solid solution after quenching whereas after treatment Q1173 most of the V was in the form of coarse VC particles formed in austenite during holding at this temperature with only a small amount retained in solid solution. Treatment FC1173 was designed to precipitate any V remaining in solid solution during the austenite/ferrite transformation. These particular heat treatments were based on the results of calculations of the VC solubility in austenite. These calculations were carried using MTDATA or ThermoCalc software. The VR steel was subjected to the Q1300 and FC1173 treatments only. All heat treatments were performed in a controlled cooling furnace or in a Theta dilatometer. The microstructure of steels A and C after treatment Q1300 were fully martensitic with a prior austenite grain-size of ~100 μ m whereas after furnace cooling from 1300K both steels had ferrite/pearlite microstructures, the remainder being martensite, resulted after quenching from 1173K.

MECHANICAL AND CORROSION TESTING RESULTS AND DISCUSSION

Mechanical Test Data

Charpy and tensile test results were obtained for the 50kg heats using standard machined test-pieces conforming to British and European standards, the results are shown in table 3. Hardness data was obtained after tempering for various times from 15minutes to 72 hours at temperatures in the range 923 to 973K. The results are presented in Figs. 1 to 3 for steels J3, J3Mo and J3CuNi. Results for J3Cu were less complete because of the rolling defects noted earlier.

Identity	Temp. C	Orientation	LYS	0.2%PS	UTS	%R/A	54J
-	_		MPa	MPa	MPa		ITT C
J3prev	675	L	n/a	639	756	n/a	-29
J3	675	Т	583	607	676	54	-90
	675	L	549	617	684	73	n/m
J3Mo	675	Т	606	683	745	75	-80
	675	L	586	677	759	72	n/m
J3Cu*	675	L	583	631	697	72	-95
J3CuNi	675	Т	616	652	711	n/a	-80
	675	L	606	659	727	n/a	n/m
J3	625	Т	747	813	899	64	-20
	625	L	788	816	886	75	n/m
J3Mo	625	Т	675	764	886	74	-15
	625	L	695	744	898	76	n/m

Table 3 Charpy and tensile testing results.

*Only 1 tensile result valid

No mechanical testing apart from hardness checks was performed on the 800 gramme melts.



Fig. 1 Tempering response of J3.



Fig. 2 Tempering response of J3Mo.



Fig. 3 Tempering response of J3CuNi

Corrosion Test Data

Corrosion testing was carried out for all steels using a test solution consisting of a mixture of NaCl and NaHCO₃ saturated with CO₂. An addition of NaCO₃ was made to the pH 8.4 tests. Additional details can be found in [6]. In all corrosion tests the pH was continually monitored throughout each test run and was controlled by the partial pressure of the CO₂, this being 1 bar for the pH 5.4 tests and 0.6 bar for the pH 8.4 testing:

Low pH 5.4: Testing at this low pH was chosen to assess the behaviour of the steels under conditions where a stable corrosion film may or may not form. The linear polarisation resistance was determined at a polarisation potential of \pm -15mV around the free corrosion potential E_{corr} after 15 minutes for a total duration of 3 to 4 hours. Data from these experiments, together with Tafel slope data obtained from a larger range in applied potential over the last 15 minutes of testing, were used to estimate corrosion rate.

High pH 8.4: This condition was chosen to reflect the increased alkalinity which prevails under the corrosion layer once this has formed. These tests were conducted starting from E_{corr} sweeping at a rate of 1mV/sec into the cathodic branch of the corrosion curve. Following completion of the cathodic branch, the potential was returned to E_{corr} and the test cell allowed to stabilise for 10 minutes before commencing the anodic sweep also at 1mV/sec.

The test temperature was maintained at 303K (30° C) throughout. In most cases duplicate test coupons were used for the corrosion tests. These results indicated that the corrosion rates were reproducible to +/-0,1mm/yr. A limited amount of testing was also carried out at 353K (80° C) for the J3 series.

To examine the corrosion behaviour under conditions more akin to service, a longer duration 'free' or open circuit corrosion potential test was performed at pH 5.4 for times up to 150 hours. Closed flow loop corrosion tests had been used previously to compare corrosion rates under conditions more akin to service but the comparisons between corrosion rate in the long term tests or LPR testing showed that the relative ranking of composition was similar to that in the flow loop tests.

The results from the LPR converted to corrosion rate in mm/yr are presented in table 4. Additional details of the method used are given in [6] but in essence involved polynomial fits of the cathodic and anodic branches of the corrosion curves then using the LeRoy and Barnett approach [9,10] to obtain Tafel slopes.

50 kg melts					
Steel Q&T	J3 prev	J3	J3Mo	J3Cu	J3CuNi
CR mm/yr	0.73	0.90	0.89	0.63	0.69
800 gramme melts					
Steel		Α	С	VR	
CR Q1300 mm/yr		1.64	0.80	1.42	
CR Q1173mm/yr		1.13	1.02	N/a	
CR FC1173 mm/yr		0.88	1.61	1.82	

Table 4 Comparison of corrosion rates.

The long term test open circuit corrosion tests and the flow loop data previously reported [1,11] provided strong evidence for the formation of stable Fe carbonate layers at the corroding surfaces. Figures 4 and 5 show a comparison between the corrosion behaviour of an X70 steel and steel C

(Fig. 5) in the open circuit corrosion experiment. The X70 data shows numerous abrupt and chaotic changes in corrosion rate which corresponded to detachment of the corrosion layer and rapid corrosion at the newly exposed metal underneath. In contrast, similar results for steel C showed fewer abrupt changes and where these were observed the rate of re-formation was slower. The differences between quenching temperatures are clearly illustrated implying significant changes in the corrosion mechanisms.



Fig. 4 Illustration of the evolution of E_{corr} with time in the long term corrosion tests.



Long term exposure for steel C as quenched



Comparison of Mechanical Properties

In general the trends on LYS, proof stress, UTS and PS/UTS ratio are consistent with the expected effects of composition. The addition of Mo from the J3 composition, J3 in this study, increases strength by ~20MPa and, as Mo increases hardenability, this effect is probably a consequence of there being a more martensitic microstructure in the quenched condition prior to tempering. On the other hand Mo also retards tempering giving a slightly higher hardness for a given heat treatment. This is illustrated by comparing Figs. 1 and 2; the Mo-bearing steel is consistently harder at a given

time at all tempering temperatures compared to the Mo free steel. The addition of 0.5% Cu to the base, Mo free, composition increases proof strength by approximately 37MPa, whereas if this were solely due to solid solution strengthening an increase of the order of 16MPa would have been expected [12]. The difference is probably attributable to a modest increase in martensite content before tempering due to the Cu addition or Cu retarding recovery rates during tempering. The combined Cu and Ni addition raises strength by slightly more than Cu alone as might be expected but again the increase is somewhat larger than would be expected from solid solution strengthening. Comparing these results with previous data from a similar Q&T steel, J3prev [1,5], showed that the results presented here for J3Mo are somewhat higher, by ~40MPa, for the same tempering treatment. The composition ranges are however not identical hence the hardenability will differ slightly. A further reason may be that both the geometry of the quenched sample and the cooling rate in the laboratory tempering treatments used in this study differ slightly from those used previously.

Although not exhaustively studied, decreasing the tempering temperature to 898K (625°C) markedly raised the 54J ITT, the cause of this is most likely to be precipitation hardening. Examination of the hardness data shows a distinct peak in the hardness at short ageing times at 898K (625°C) which broadens somewhat in the Mo and CuNi steels. Unfortunately, due to the presence of rolling defects, insufficient hardness data was obtained for the Cu containing steel although it is clear that the peak hardness at short tempering times for the CuNi steel is slightly lower consistent with the known effect of Cu delaying VC precipitation [13,14]. Additionally, the broader hardening range displayed by J3CuNi suggests that CuNi additions delay VC precipitation to longer times, for example after 180mins at 948K (650°C) the hardness values are 236, 258 and 270 for J3, J3Mo and J3CuNi, respectively.

Comparison of Corrosion Behaviour

The extent of the improvement in corrosion behaviour accruing from the alloying strategy devised previously can be judged from results on X65 and X70 subjected to flow loop testing at IFE (Norway), corrosion rates ranging from 8.3 (X65) to 6.0 (X70) mm/yr. The previous study which used Cr additions together with V, Mo and Cu additions demonstrated that this range could be reduced and typically gave values in the range 0.6 to 3mm/yr [1] under the same conditions. In addition, the previous study [1] also showed that the corrosion rate inferred from the LPR measurements gave the same relative ranking as the flow loop tests, X70 giving approximately twice the corrosion rate as J3prev, 1.48 compared to 0.73mm/yr. The LPR corrosion data for the J3 series in this study confirmed that Si, Cu and Ni additions modify or improve corrosion resistance in carbonic acid. The Si content in the J3prev composition was significantly higher than in the study presented here and the corrosion rate was correspondingly lower than in the steels tested here, 0.73 mm/yr for J3prev compared to 0.9mm/yr for J3. In contrast, increasing the Cu content to 0.5% decreased the corrosion rate, 0.63 for J3Cu compared to 0.9 for J3. On the other hand adding Ni did not reduce the corrosion rate further, J3CuNi being similar to J3Cu, 0.69 compared to 0.63 mm/yr. Increasing the test temperature to 353K (80°C) increased corrosion rate to ~4-5mm/yr for J3, J3Cu and J3CuNi, but the increase was noticeably greater for J3Mo, ~6.5mm/yr at the end of the test period.

The most interesting results were obtained for the 800 gramme series designed to examine the effect of V. For steel A, V-free, the corrosion rates lay between 0.88 and 1.64mm/yr depending on the metallurgical heat treatment whereas steel C containing V gave values in the range 0.8 to 1.61mm/yr. Not only did the corrosion rate differ because of the V addition but there is also an effect of microstructure. There were marked differences in corrosion rate for steel A probably because of the different microstructures formed after heat treatment. Furnace cooling from 1173K

(ferrite/pearlite microstructure) gave a value of 0.88mm/yr compared to 1.12mm/yr after quenching from the same temperature (bainitic microstructure) which in turn was lower than that obtained after quenching from 1300K (martensitic microstructure). There was a significant reduction in corrosion rate after quenching from 1300K when V is added, 1.64 compared to 0.8mm/yr. These observations suggest that because the microstructures of the two steels were similar after the 1300K heat treatments, both were predominantly martensitic, the presence of V has had a marked effect on corrosion processes. Making the assumption for heat treatment Q1173 that the microstructures are similar and any differences did not influence corrosion rate, the corrosion rates are only marginally affected by V at least within the errors of measuring corrosion rate. The different behaviour can be rationalised in terms of the metallurgical state of the V addition. After treatment Q1300, thermodynamic calculations indicate that all the V is retained in solid solution in austenite whereas after quenching from 1173K would cause some VC to form during the austenite to ferrite transformation the particle size will be significantly coarser than that after the Q1173 condition and it would appear that the corrosion rate rises significantly.

It can therefore be concluded that V modifies the corrosion processes occurring within the corrosion layer and thereby reduce corrosion. The degree of protection appears to depend, from these results, on both the base microstructure and whether V is in solid solution or not. It is also possible to interpret the data in terms of VC particles accelerating corrosion. Neither of these issues has been proven by this study but it was clear from examination of the corrosion layers on the Q1300 sample that there is V enrichment as well as Cr (Fig. 6). It has not been possible to draw any conclusions from examination of the corrosion layers on FC1173 because of the extreme fragility of the layers.



Fig. 6 Secondary electron and back scattered SEM images, SE and BSE, compared to element X-ray maps for C, V, Cr and Fe. Note the concentration of V and Cr in the corrosion layer.

A somewhat more definitive effect of microstructure can be noted in the LPR corrosion results from the VR (C free) steel in which virtually all the V, ~0.6%, remains in solid solution throughout processing. These data are shown in Fig. 7 and demonstrate that the martensitic structure formed after quenching from 1300K corrodes less quickly than the ferritic microstructure formed after furnace cooling from 1173K but the initial corrosion rate is about half of that for the ferritic microstructure, ~1.4mm/yr compared to ~3.6mm/yr. If this effect of microstructure is similar for steels A and C then it might be anticipated that the increased corrosion rate noted for steel C after furnace cooling could largely be attributed to the presence of VC precipitates. The decrease in corrosion rate in the early stages of the test may be linked to the build-up of V in the carbonate rich corrosion layer as shown in Fig. 5. Furthermore, it is possible that there are differences in the rate at which V becomes incorporated in the corrosion layers because of the different chemical states of V in the steel matrix and hence these are also reflected in changes to the corrosion rate.





Suggestions for Further Steel Development

There are some practical implications of the work presented here. The aim of producing an X80 to X100 (550 to 690MPa) steel with better corrosion resistance is close to being realised for an X80 (550MPa) steel, the J3CuNi composition achieves X80 after tempering at 948K (675°C) but not the J3 steel. Compared to the same steel with higher Si, J3prev, the difference in strength is substantial, J3prev attained in excess of X90 (620MPa) in the quenched and tempered condition although Charpy toughness was disappointing [1]. The results of the tempering studies provide further ideas for improving the J3 composition. It seems clear that Cu additions delay V precipitation, this finding duplicates earlier Japanese work on plain C steels and this does not appear to have had a detrimental effect on Charpy toughness. However, tempering at 898K (625°C) raises strength considerably, upto X110 (~780MPa), but Charpy toughness decreases dramatically from around -80C to close to -20C. This decrease is consistent with the precipitation hardening that can be inferred from the tempering curves, that for J3 shows a distinct peak in the region of the tempering times used for the mechanical tests. Given that Cu additions broaden the V hardening response then it may be possible to exploit this by changing the tempering treatment to optimise both the tensile and Charpy properties or, by increasing the Cu addition. However, the experience in this study is that Cu alone would create processing problems associated with hot shortness. Nevertheless, by judicious use of Ni additions and control of furnace atmosphere composition during processing, this problem may not be insuperable. Increasing Si content would also modify the tempering response, increasing strength closer to the X100 target, but may lead to problems with casting and adherent scale formation.

All of the compositional changes outlined above would improve corrosion resistance to carbonic acid. However, the most interesting result is the finding that V in solid solution but probably not as relatively coarse precipitates is responsible for improved corrosion resistance in the media tested. It would therefore be of great interest to examine whether the corrosion resistance might be increased further by larger V additions. To a degree this possibility was assessed in the C-free steel, VR,

where virtually all the V is present in solid solution. The corrosion rate in this instance was greater than that of steel C in which the V was present either in solution or as fine particles, heat treatment Q1300. That steel C showed better corrosion resistance than the VR steel might suggest that part of the protective effect of V arises through the latter but this has not been confirmed by electron microscopical studies. Comparison with other steels, albeit without Cr additions, suggest that at such a cooling rate part of the V would be present as fine coherent VC particles [14,15]. Clearly further work into the role of V in controlling corrosion is justified particularly because there is a clear need to further optimise the mechanical properties by modifying both Cu and Ni additions and tempering treatments. Whether or not reducing the Cr addition to ~2.5% and raising the V content to ~0.5% would produce any improvements to corrosion behaviour is also worth exploring.

CONCLUSIONS

1. The results of a study examining the effects of Si, Mo, Cu and Ni on the mechanical properties and corrosion rate of Q&T steels designed for improved corrosion resistance in carbonic acid, pH 5.4, has demonstrated that further improvements can be made. The extent of improvement is dictated by the tempering treatment, although strength levels upto X100 are possible with this type of steel further work is necessary to achieve the desired Charpy impact properties.

2. Mo and Cu additions have been shown to inprove mechanical properties and reduce corrosion rate. The Si content of the steels in this study is lower that that assessed previously but this study has confirmed the beneficial effect of increasing Si content on the corrosion rate in carbonic acid at a pH of 5.4.

3. By comparing the results from steels in different metallurgical conditions, a significant improvement in corrosion rate can be attributed to the presence of V. The present results are consistent with V being most effective if present either in solid solution or as a dispersion of fine precipitates. Further work may lead to a better definition of the metallurgical role of V and opens up the possibility of deliberately alloying with combinations of Cr and V to further improve corrosion behaviour of the novel class of steels.

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