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Variation in Mechanical Properties and Corrosion Resistance of Different Alloys within the Generic Designation UNS S32760

G. Byrne, R. Francis & G. Warburton
(Weir Materials and Foundries UK)

Abstract

Zeron 100 super duplex stainless steel was first developed in the 1980's and it was invented with a tightly controlled melt chemistry which together with a restricted heat treatment temperature range ensured the optimum combination of mechanical properties, corrosion resistance, weldability and hot working characteristics. When the alloy was included in ASTM, ASME, BS, EN, NACE and other standards, Zeron 100 data was submitted whilst the alloy was eventually listed under the UNS S32760 designation. UNS S32760 however, has a much wider range of chemical composition than Zeron 100. During the 1990's this grade of super duplex stainless steel became very popular with the oil and gas industry for both sea water and process systems. This commercial demand has resulted in the S32760 grade being offered by an increasing number of alloy producers. However, the pressure to minimise production costs has resulted in some manufacturers pushing the composition to the limits permitted by the UNS specification. This can give unusual combinations of analysis and shows a lack of fundamental understanding of the required control in composition and thermo-mechanical processing.

For example, nickel levels have been reduced and nitrogen contents have been increased, together with hot working and final heat treatment disciplines that are variable. These variables are not directly apparent from material certification. There are many examples of the problems that this causes, where commercial heats of S32760 exhibit worse than expected properties. The paper describes a series of laboratory tests on several commercial heats of S32760 to illustrate this point.

One effect which can result is the precipitation of nitrides. In an attempt to understand the effects of the poor control on precipitation behaviour an arbitrary nitirdes scale was developed which assigned a number from 0 to 3 based on an examination of a large number of fields on a microsection. Evaluation of impact toughness showed a sharp reduction in toughness with increasing nitride content. Corrosion tests also showed a substantial reduction in corrosion resistance with increasing nitride content.

These effects are discussed in the context of industry using material that does not meet the properties that were submitted originally to enter the grade into international standards.

1. Introduction

The first widely available super duplex stainless steel was developed by Gradwell and co workers (ref 1) in the mid 1980's. This alloy was called Zeron 100 ® and was developed as a casting alloy for pump applications in the oil and gas industry. The performance of the steel in this application generated a demand for Zeron 100 in wrought product forms also. This demand was serviced by Weir who, together with other European manufacturers, developed the manufacturing procedures required to obtain the desired quality of product.

As demand for the steel grew, clients called for ASTM, NACE, British Standards and other codes to include and cover the Zeron range of products. Moreover, with the introduction of the EC Procurement Directive in 1993, it became illegal for clients to specify any trade name in requests for quotation and a generic description for Zeron 100 was required for the business to continue.

In 1993 - 94 ASTM considered the properties of several heats of Zeron 100 in a range of product forms and on the basis of this designated the code UNS S32760 to the alloy and introduced this number into several standards.

In 1994 UNS S32760 was included in NACE MR0175, again based upon the performance and properties of Zeron 100. Finally in 1997 UNS S32760 was listed in ASME complete with applicable design stresses. These were again based upon the properties of Zeron 100.

Subsequently, other steelmakers began to manufacture UNS S32760 and to offer this grade to the market. During the period of manufacture and deployment of these steels it became apparent that the properties and performance normally expected was not always realised. The properties which began to deteriorate included elevated temperature tensile properties, low temperature toughness properties and, in Norway, performance in ferric chloride (ASTM G48 Method A) at 50°C. Similar behaviour was also being experienced by UNS S32750, which is also a super duplex stainless steel. The variation in performance seemed intermittent and ad-hoc. This behaviour has never, to our knowledge, been systematically researched because it was often encountered on a production basis and was accepted or rejected on the strength of engineering critical assessment and the problem was generally never revisited.

This paper details the results of an exercise where UNS S32760 bar product, to ASTM A276, was sourced from the market and tested.

Whilst all the material sourced complied with the UNS S32760 specification range in all respects, its performance in corrosion tests and also, in Charpy impact tests, was highly variable.

However, whilst these products generally met normal oil industry specification requirements, the margins by which the product exceeded the acceptance requirements were, in some cases, minimal. Moreover, when properties like resistance to hydrogen embrittlement was examined (which is not normally a prescribed requirement in specifications, but rather, it is assumed that if specification requirements are met then an acceptable resistance to hydrogen embrittlement would be attained) it was found that this did not always give the expected results.

2. Materials

A range of suppliers were sourced and bar products purchased. The size of the bars and the chemical analysis provided with the material certificates is given in Table 1.

Further chemical analysis using spectrographic standards calibrated with respect to 25% chrome super duplex alloys is given in Table 2.

These casts were selected for evaluation because of the range of microstructure that they exhibited in so far as the nitride content was concerned. Photomicrographs are shown in Figures 1 to 3 and the results of quantitative metallography carried out to further characterise the structures are given in Table 3.

3. Experimental Method

The following equipment, techniques and standards were employed in the evaluation.

3.1 Metallography

All the samples cut for metallography were large (typically 10-15 cm^2) to show as wide a variation in the microstructure as possible.

Transverse samples of each bar were cut, polished to 1μm diamond finish and double etched electrolytically in oxalic acid followed by potassium hydroxide. The samples were typically etched at 3.5V, 0.3A for 5-10 seconds in oxalic acid and 1 to 2 seconds in potassium hydroxide at the same settings.

The phase balance was measured on 10 fields using an image analysis technique with a typical error of ± 5%.

The interphase spacing was measured using a calibrated graticule and a computer generated tape. The spacing was measured 70 to 100 times for each microsection and the mean and standard deviation were calculated. There was considerable variation in the interphase spacing, as shown by the large standard deviations in Table 3.

In order to determine the effect of nitrides it was necessary to find a way of assessing the quantity of nitrides present in each sample. There is no standard method for doing this and so an arbitrary nitride scale was developed.

Nitride No.
0 - No nitrides visible
1 - Some ferrite grain boundaries visible but not as an extended network.
2 - Substantial amount of ferrite grain boundaries visible, forming a network in many places. A few dark precipitates within the grains.
3 - Extensive ferrite grain boundary network and large quantities of precipitates within the grains.

It was found that the nitrides were best observed after polishing to 1µm followed by an electrolytic etch in oxalic acid alone. In order to get a reliable assessment of the nitride number it was necessary to examine a large number of fields at X500 to obtain an overall impression of the condition of the sample. In some cases the fields showed a roughly equal division between two nitride numbers and then they were given an intermediate value. The nitride numbers for the six alloys under consideration were:-

OC9943 - 3
OC11008 - 0
OC10210 - 1½
OC10211 - 2
OC10212 - ½
OC10213 - 1

No other precipitates, e.g. sigma, were seen in any of the samples.

3.2 Charpy Impact Tests

Standard Charpy vee notch impact test specimens were taken in accordance with ASTM A370. These specimens were orientated longitudinally down the bars with the notch in the transverse direction. Tests were carried out over a range of temperatures to produce a transition curve for each material.

3.3 Corrosion Tests

Two corrosion tests were carried out, the first of which was a critical pitting temperature (CPT) test in 10% hydrated ferric chloride solution (ASTM G48). The sample faces were prepared to 120 grit and the sharp edges were bevelled. The samples were exposed initially for 24 hours at 50°C and then removed, washed, dried and weighed. If no significant weight loss was observed the samples were tested for a further 24 hours at 5°C greater temperature, and so on until pitting occurred.

Sample OC9943 failed at 50°C and so a further test was conducted with this material starting at 40°C. All alloys were tested in duplicate.

The critical pitting temperature (CPT) at +600 mV SCE in synthetic sea water was also determined. This potential was chosen because it is typical of that observed for high alloyed stainless steels in chlorinated sea water.

In this test the sample is polarised to +600 mV SCE over 30 minutes at 20°C and the current is allowed to stabilise for one hour. The temperature is then increased at 5°C/hour until a large increase in the current density shows that pitting has initiated. The CPT is that temperature at which the current density reaches $10\mu A/cm^2$.

3.4 Hydrogen Embrittlement Tests

Slow strain rate tests were conducted at a strain rate of 1×10^{-6}/sec to compare the resistance to hydrogen embrittlement. Tensile specimens were machined in the direction of rolling of the bar i.e. longitudinally, with a gauge length of 30mm and a cross sectional area of $30mm^2$.

One sample was tested at ambient temperature in air, while a second was placed in a cell containing synthetic sea water and polarised to -1.04 V SCE. The sea water was deaerated and 5mg/l sulphide was added as sodium sulphide solution, adjusted to pH8, to poison the hydrogen recombination reaction. The loss of ducticity resulting from hydrogen charging was evaluated using the plastic strain ratio, as described in EFC publication No. 17.

In addition two alloys were tested at a series of constant loads, while polarised to –1.04 V SCE in sea water to determine the threshold stress. This test method is very time consuming and so only tests representing no nitride precipitates and a high level of nitride precipitates (level 3) were chosen (OC11008 and OC9943 respectively). Samples which had not failed after 14 days were removed for examination.

4. Results and Discussion

4.1 Chemical Analysis

Comparison of the chemical analysis given on test certification compared with the results from analysis against calibrated standards (Tables 1 and 2) showed, for the alloys supplied, that test certificates generally overestimated the level of nickel in the steel and underestimated the nitrogen content. This gives a Ni/N ratio which is artificially high, and the smaller the Ni/N ratio, the greater the tendency for nitride precipitation would be expected to be.

4.2 Microstructures

All the samples exhibited a duplex structure with grains elongated in the rolling direction of the bar. The samples differed in the extent and degree of nitride precipitate and level of reformed austenite (reformed austenite can be identified by its Widmanstätten type appearance and/or because unlike the primary austenite and ferrite, it is not elongated in the rolling direction of the bar. Where the level of nitride precipitate is high, this then corresponds to a lack of reformed austenite and therefore a larger interphase spacing as shown in Table 3 and Figures 1 to 3).

The phase balance of the samples varied between 44 and 57% ferrite (i.e. 50% ± 7%). Figure 4 is a graph of phase balance plotted against nitride number. This graph shows no clear trend between the variables except perhaps that overall ferrite contents increase with increasing nitride number. This behaviour can be reconciled if it is understood that nitrogen in solid solution promotes austenite formation. Therefore, when nitrogen is tied up in the form of a nitride its influence as an austenite forming element is lost. However, the range of ferrite contents measured in these bars is quite narrow and well within normal specification requirements.

4.3 Charpy Impact Behaviour

Figure 5 shows the Charpy impact energy achieved at –46°C. Since these alloys are often used in heavy wall thickness, for components carrying fluids at high temperature and pressure, the possibility of sudden depressurisation and chilling of the component down to temperatures of the order of –40°C and below exists.

Hence, the material must have sufficient toughness to resist fracture and leak before it breaks. For duplex stainless steels, most specification requirements call for impact energy requirements in the range 40 to 70 joules as an average of three samples.

From Figure 5 it is clear that increasing nitride number significantly reduces the absorbed energy to levels which only just exceed the minimum acceptance level of some specifications.

Fractography of the test specimens without nitrides revealed fully fibrous, ductile failure mode, with pronounced shear lips, at test temperatures down to –60°C. Scanning electron microscopy confirmed the ductile failure mode (microvoid coalescence). Samples with nitride level 3, even when tested at –20°C, exhibited a rather flat fracture face, with crystalline facets and narrow shear lips. The extent and degree of crystallinity increased with decreasing test temperature. Scanning electron microscopy showed mixed mode failure i.e. ductile (microvoid coalescence) and areas of quasi cleavage facets. Measurements of lateral expansion from samples tested at – 40°C and –60°C are shown in Figure 6. From this figure it is clear that heavily nitrided material always gives poor notch ductility whilst materials with zero or low levels of nitride always give good notch ductility results. However, at intermediate nitride levels a reduction in test temperature of –20°C reduces the lateral expansion by about 60%.

Figure 7 shows the temperature at which specimens give an impact energy of 150J average (i.e. the 150J transition temperature). With zero and low nitride level samples this occurs at –80°C to –90°C and progressively deteriorates with increasing nitride content until at nitride level 3 the 150J transition temperature occurs close to 0°C. This is a 90°C shift in performance level.

The overall deterioration in performance with increasing nitride level is of concern because bar product is often transformed by forging or other hot working processes which may lead to further deterioration in properties. Hence if the properties of the base material are not as high as anticipated, the margin for in-process reduction in properties is reduced, and intermittent or more frequent failure to meet specification requirements can be encountered. Furthermore, because the nitrides are extremely stable once formed, attempts to apply remedial heat treatments in an attempt to dissolve these precipitates can prove futile.

4.4 Corrosion Test Results

4.4.1 ASTM G48 ferric chloride test results. Figure 8 shows how the critical pitting temperature (CPT) in ferric chloride varies with nitride content. Up to a nitride level of about 1.5 there is no discernible effect of nitrides on CPT. Above 1.5 nitride level the CPT falls progressively until at level 3 a CPT of 45°C is reached.

The significance of the 45°C CPT is that in the UK specifications call for no pitting at 40°C in ferric chloride, whilst in Norway, Norsok standards require a 50°C CPT. This means that nitride level 3 materials would be accepted for an offshore development on the UK continental shelf but would be unacceptable in the Norwegian sector.

Figure 9 shows a microsection through a ferric chloride test sample. Corrosion can be seen to initiate at nitride precipitates within ferrite grains.

4.4.2 CPT evaluations in synthetic sea water mirror the behaviour in ferric chloride as shown in Figure 10. The lowest CPT achieved was 62°C at nitride level 3. This temperature is significantly higher than that at which most chlorinated seawater systems operate on offshore platforms. However, it is known (ref 2) that short and long term temperature transients exceeding 68°C can occur. In these circumstances it is clear that nitride level 3 material would probably have suffered crevice corrosion where field experience has shown that prime quality material is able to tolerate these conditions without corrosion attack (ref 3).

4.5 Resistance to Hydrogen Embrittlement

Previous work in this area by Weir (Ref 4), our competitors (Ref 5) and independent university research (Ref 6) have all shown that threshold stresses for the onset of hydrogen embrittlement as a consequence of cathodic protection of super duplex stainless steels is of the order of the specified minimum UTS of the material (750 MPa). Work on nitride level 3 material has shown that this threshold stress is reduced to the yield strength of the material (580 MPa) as shown in Figure 11. This unexpected reduction in performance is attributed to the presence of nitrides. Recent experience in the North Sea (ref 7) has highlighted the unexpected failure of large super duplex stainless steel forgings due to hydrogen embrittlement as a consequence of cathodic protection. Many factors were implicated, such as poor or no insulation coating, cathodic overprotection, poor design, machining across the grain flow within the forging presenting a longitudinal rather than a transverse grain flow to the environment and also, critically, the presence of high applied and residual stresses within the structure. However, it is also true that the forgings contained nitrides and that these may have played some role in the problem.

Slow strain rate investigations have shown that the plastic strain ratio falls from 60% to 20% as nitride contents go from 0 to level 3, as shown in Figure 12 i.e. a 40% loss in performance level.

Figure 13 plots the plastic strain ratio for nitride level 3, level 0.5 and level 0 materials against applied potential. From this graph it appears as though the presence of nitrides sensitises the materials to hydrogen embrittlement. Whilst only level 3 material shows sensitivity (i.e. loss of performance) at –700 mV SCE it is clear that as the potential becomes more negative the performance of material with and without nitrides diverges, with nitrided material performing poorly.

Figure 14 is a micrograph of secondary cracking from a level 3 sample exposed to –1.04V SCE. This seems to show that the cracking is associated with the nitride precipitates. When specimens with lower nitride levels are examined in the same way the correspondence is not so clear.

The present work has only investigated the effect of nitrides on corrosion resistance in two common fluids, ferric chloride solution and sea water. There remains a wide range of other fluids where super duplex stainless steels are regularly used e.g. acids, alkalis etc, and where nitrided material may give inferior performance.

4.6 General Discussion

To our knowledge the first people to experience unexpectedly low properties in super duplex stainless steel components were a UK oil and gas engineering company, circa 1994. Their response was to impose limitations upon the supply industry on nitride-forming elements like aluminium, vanadium, and niobium, which were quickly found to be impractical. As the number of steelmakers producing UNS S32760 increased ad-hoc, incidents of failure to meet expected properties became more frequent, but the problem was never addressed directly and in fact continues to this day.

It should also be understood that some nitride precipitation is unavoidable since the ‘C’ curve for nitrides intersects the time axis (ref 10). Until recent years the nitrides remained largely sub microscopic, and, therefore, rarely observed.

Nitrides can also be encountered in weldments, and it is interesting to remember that in the early days of lean 22% chromium duplex stainless steel, highly ferritic HAZ structures, often containing nitrides, gave poor properties. This issue was resolved to a large extent by increasing the nitrogen content of the base material to 0.15% minimum (ref 8) and controlling the weld thermal cycle such that back diffusion of chromium from the matrix replenished the chromium depleted zone around the chromium nitride precipitate (ref 9). This process probably relieved tessellated stresses around these particles also.

Hence, the welding engineer controlled base chemistry, phase balance and thermo-mechanical processing of the joint in order to improve properties which corresponded with a much reduced nitride level and/ or a rather different nitride precipitate.

Our experience has been that if the alloy chemistry is imbalanced, then it is extremely difficult to avoid significant nitride precipitates irrespective of thermomechanical processing. With good chemical balance, control of thermal history is still required if nitride precipitation is to be minimised and its negative effect eliminated.

In order to avoid the problem in wrought products it is necessary to properly balance the alloy chemistry of the steel along with the phase balance and the thermal cycles experienced by the steel during processing (i.e. not only the final solution heat treatment regime), just as in welding.

The production problems encountered are of relatively recent vintage and are common to both UNS S32760, S32750 and possibly other super duplex grades. Another feature of the phenomenon is that the steel in question invariably is not manufactured by the steelmaker involved in the original development of the alloy.

The trend towards steel makers simply making to a recipe given in ASTM standards and quoting a UNS reference number seems inappropriate for these more sophisticated alloys, as evidenced by the nitride problems. The performance based acceptance of alloys into codes like NACE MR0175 and pressure vessel codes should be specific to the alloy under consideration (sources and manufacturing route). Blanket approval of generic designations of sophisticated materials may be flawed.

It is recommended that users carefully qualify vendors of sophisticated materials and disqualify those vendors without the necessary detailed back up information. Where new or novel equipment is being sourced in sophisticated materials or the periphery of manufacturing experience is being tested by project demands, it is recommended that the user utilises the original source of the materials of construction of the item and that both user and supplier discuss and understand where problems may lie and how they may be resolved. In such cases a prototype approach is recommended.

5. <u>Conclusions</u>

5.1 The presence of nitride precipitates in UNS S32760 reduces Charpy impact performance. Increasing levels of precipitate lead to lower toughness levels, lower notch ductility and an increasingly brittle fracture mode.

Differences in performance between material containing nitride precipitates and that without nitride precipitates become enhanced at lower test temperatures.

5.2 Corrosion resistance in ferric chloride and also in synthetic sea water, as demonstrated by critical pitting temperature measurements, does not deteriorate until significant levels of nitride precipitates are present. Both environments showed the same trend in behaviour. No deterioration in performance was observed until nitride level 1.5 had been exceeded and then a significant fall in CPT was measured.

5.3 Nitride precipitates reduce the resistance of the material to hydrogen embrittlement. This is demonstrated in the form of reduced threshold stresses for the onset of cracking from 750 MPa to 580 MPa and also in slow strain rate testing by a reduction in the plastic strain ratio. In these tests a 40% deterioration in performance was measured at a potential of –1.04V SCE.

5.4 Since normal specification properties were met at all nitride levels, it cannot be assumed that this necessarily implies that other properties are as expected. This is evidenced by the lower than expected resistance to hydrogen embrittlement in these tests.

5.5 This phenomenon is common to UNS S32760 and UNS S32750 (and probably other super duplex grades).

The problem has gradually become apparent over recent years and relates to supply from steelmakers not involved in the original development of the alloys and who are supplying to the generic UNS designation rather than to their own developed and researched analysis and thermal history.

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TABLE 1. Composition of samples taken from mill certificates.

OC NO.	BAR DIA.	COMPOSITION (Wt%)											PREN*
		C	Cr	Ni	Mo	Cu	W	N	Si	Mn	P	S	
9943	160mm	0.023	25.6	6.95	3.57	0.59	0.67	0.23	0.38	0.67	0.028	0.000	41.0
11008	0.5”	0.017	25.34	7.02	3.56	0.66	0.53	0.23	0.16	0.48	0.030	0.001	40.8
10210	4.5”	0.019	25.20	6.73	3.38	0.60	0.60	0.25	0.44	0.59	0.025	0.001	40.4
10211	160mm	0.020	25.42	7.05	3.60	0.74	0.69	0.25	0.36	0.50	0.022	0.000	41.3
10212	4.5”	0.016	25.22	7.12	3.60	0.56	0.58	0.21	0.26	0.47	0.023	0.001	40.5
10213	4.5”	0.024	25.38	7.06	3.52	0.64	0.63	0.21	0.29	0.61	0.029	0.001	40.3

* PREN = % Cr + 3.3% Mo + 16% N

TABLE 2. Composition of the samples as determined by analysis at WM&F

OC NO.	BAR DIA.	COMPOSITION (Wt%)											PREN*
		C	Cr	Ni	Mo	Cu	W	N	Si	Mn	P	S	
9943	160mm	0.022	24.9	6.95	3.58	0.56	0.66	0.25	0.25	0.58	0.021	0.004	40.7
11008	0.5”	0.020	25.2	6.95	3.60	0.63	0.57	0.24	0.18	0.48	0.028	0.004	40.9
10210	4.5”	0.016	25.1	6.83	3.36	0.63	0.72	0.26	0.46	0.58	0.023	0.004	40.3
10211	160mm	0.025	25.2	6.9	3.64	0.73	0.72	0.28	0.33	0.49	0.020	0.004	41.7
10212	4.5”	0.019	25.2	7.04	3.63	0.52	0.62	0.24	0.27	0.46	0.023	0.004	41.0
10213	4.5”	0.026	25.5	6.77	3.60	0.59	0.65	0.22	0.30	0.59	0.023	0.004	40.9

* PREN = % Cr + 3.3% Mo + 16% N

TABLE 3. Results of quantitative metallography carried out at WM&F

OC NO.	BAR DIAMETER	ARBITRARY NITRIDE NO.	FERRITE CONTENT (%)	FERRITE SPACING	
				SPACING (µm)	STD. DEV.
9943	160mm	3	56.1	35.6	27.5
11008	0.5”	0	52.0	7.1	5.5
10210	4.5”	1½	43.9	25.7	20.5
10211	160mm	2	47.9	22.4	12.7
10212	4.5”	½	57.5	15.2	10.1
10213	4.5”	1	56.0	37.9	32.8

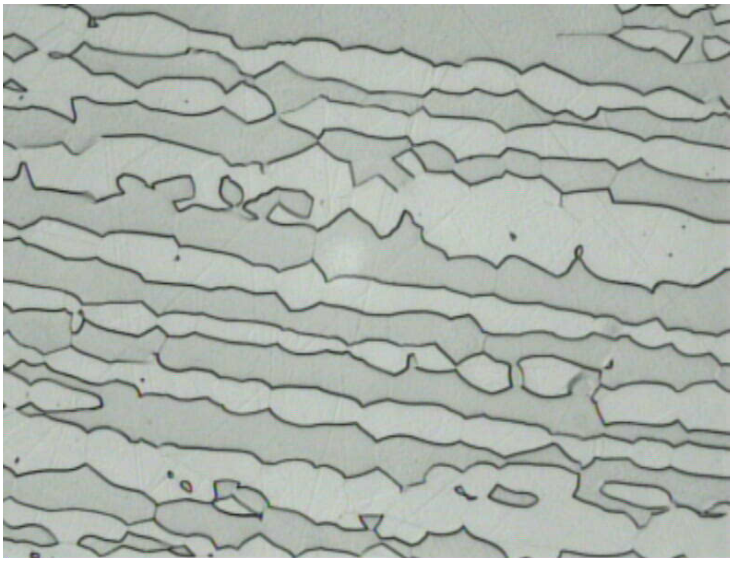


FIGURE 1 A typical microsection of sample OC11008 (nitride number 0) [X500]

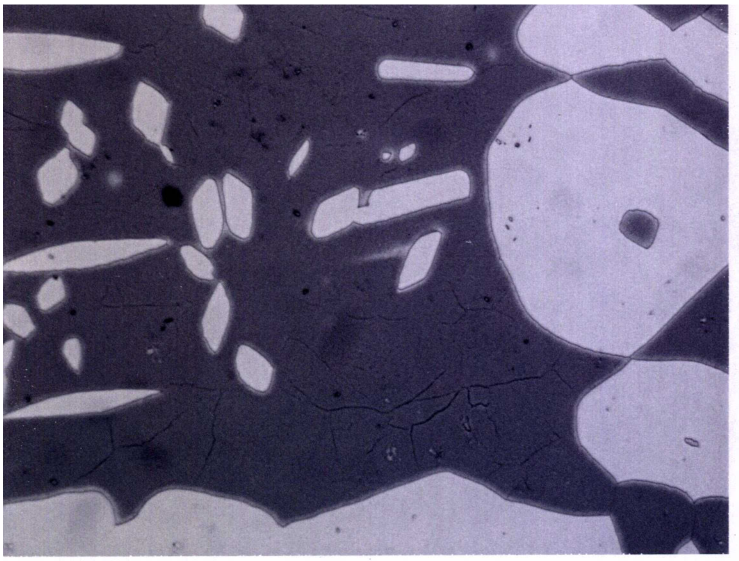


FIGURE 2 A typical microsection of sample OC10210 (nitride number 1.5) [X500]

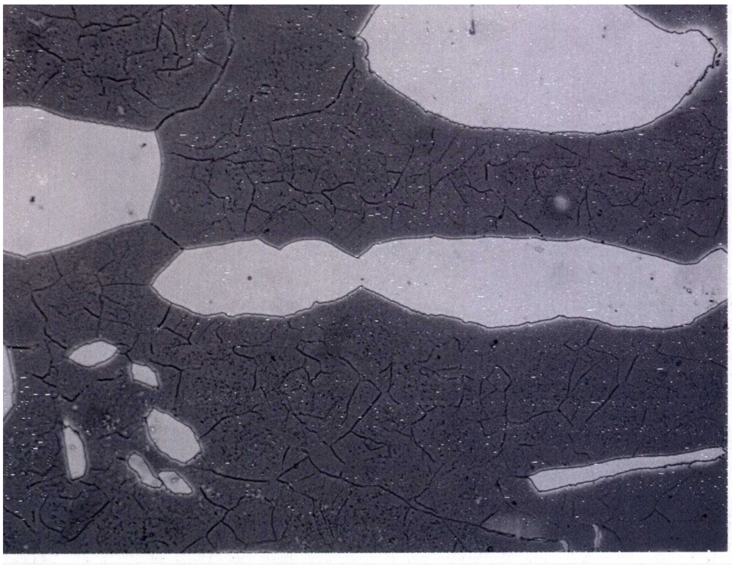


FIGURE 3 A typical microsection of sample OC9943 (nitride number 3) [X500]

FIGURE 4 Phase balance versus nitride number

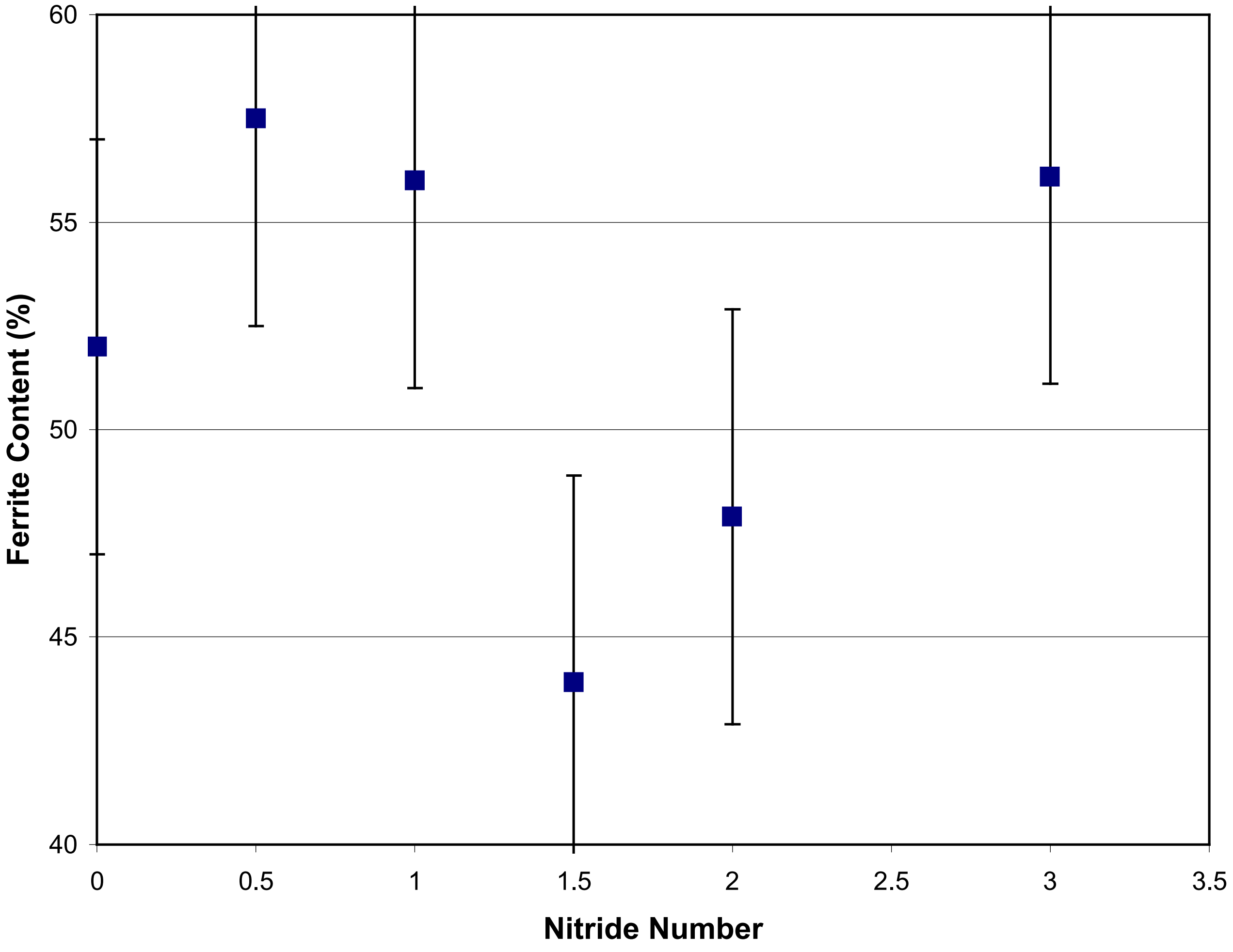


FIGURE 5 Impact toughness energy at -46°C versus nitride number

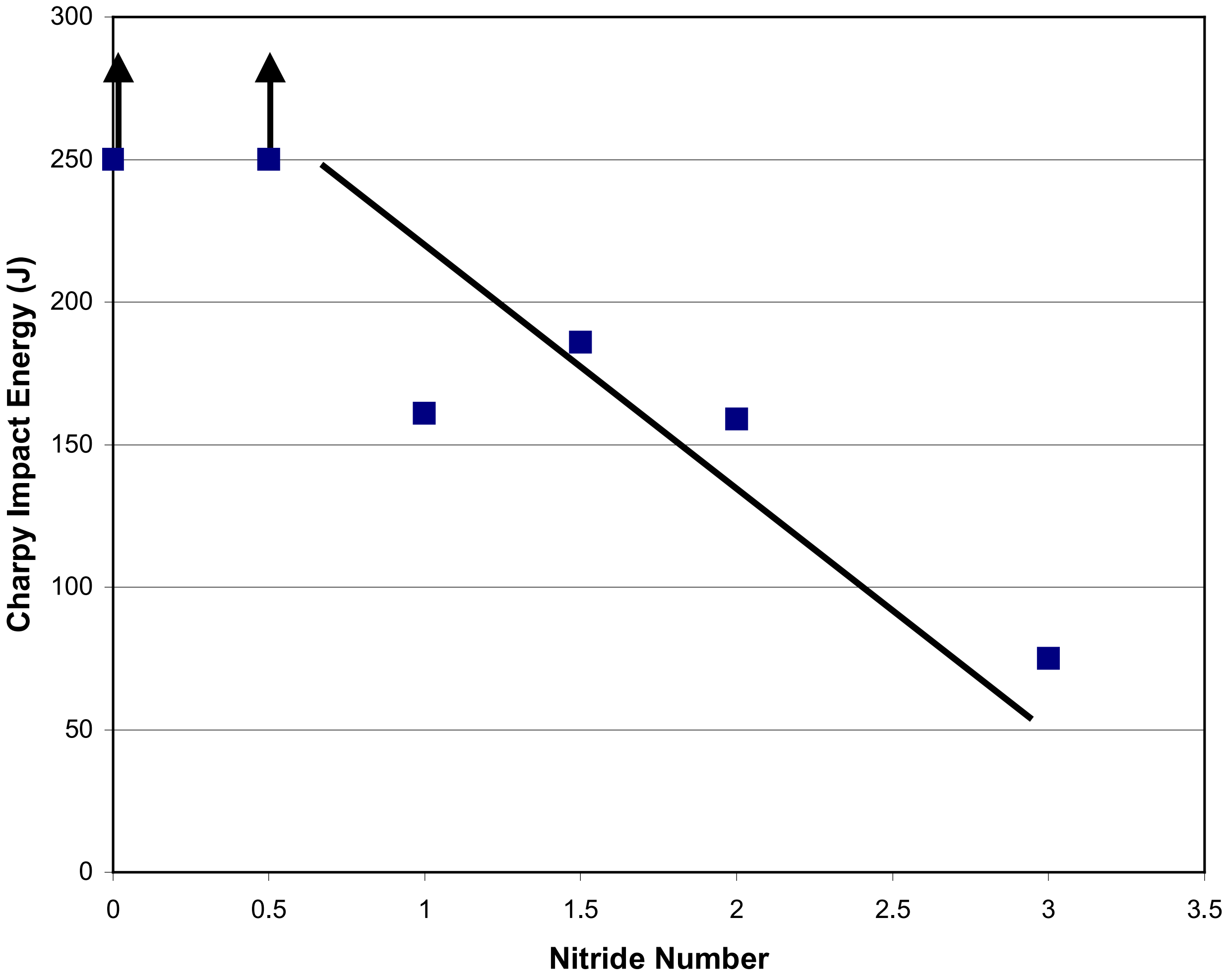


FIGURE 6 Lateral expansion of Charpy samples versus nitride number

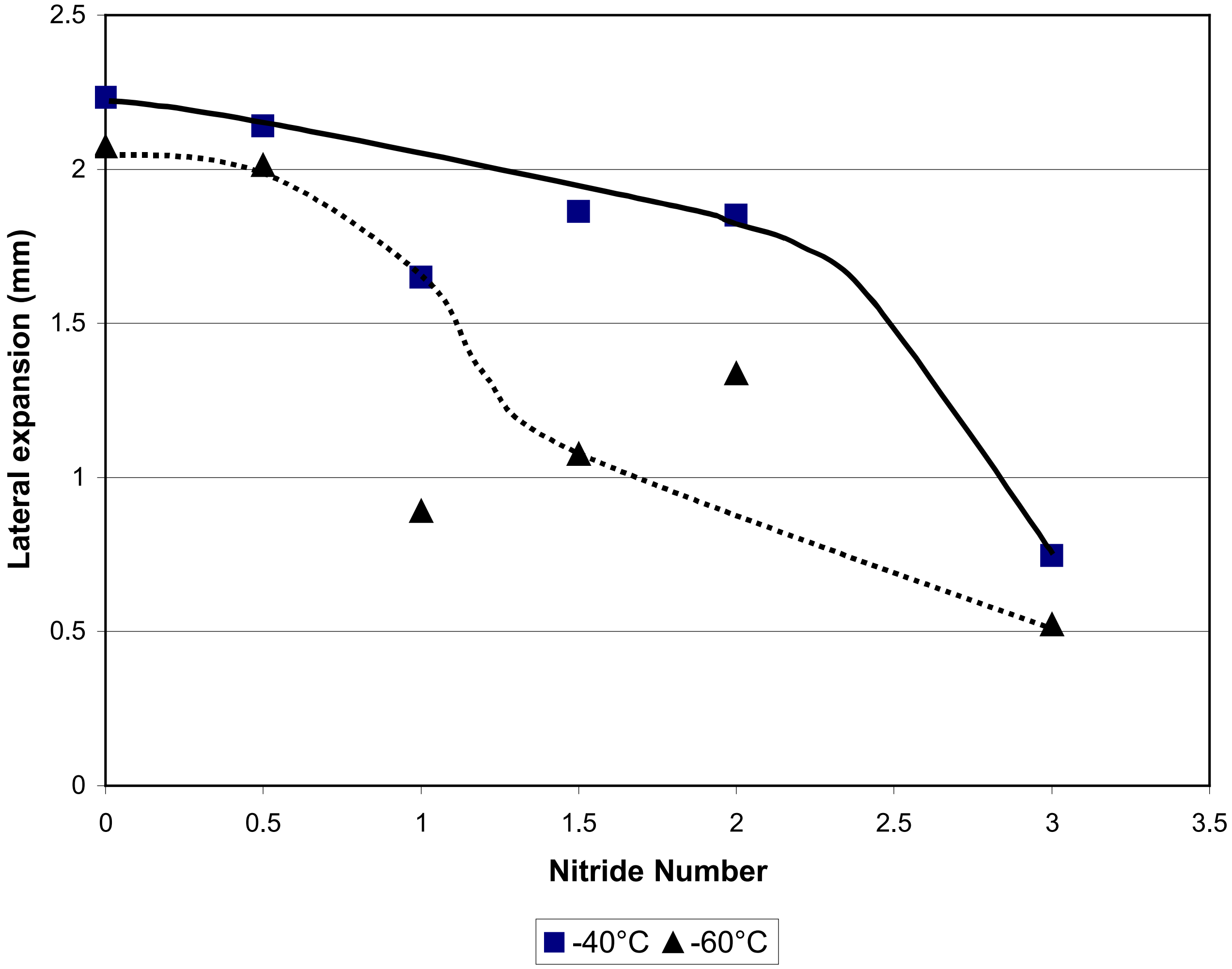


FIGURE 7 Charpy transition temperature (150J) versus nitride number

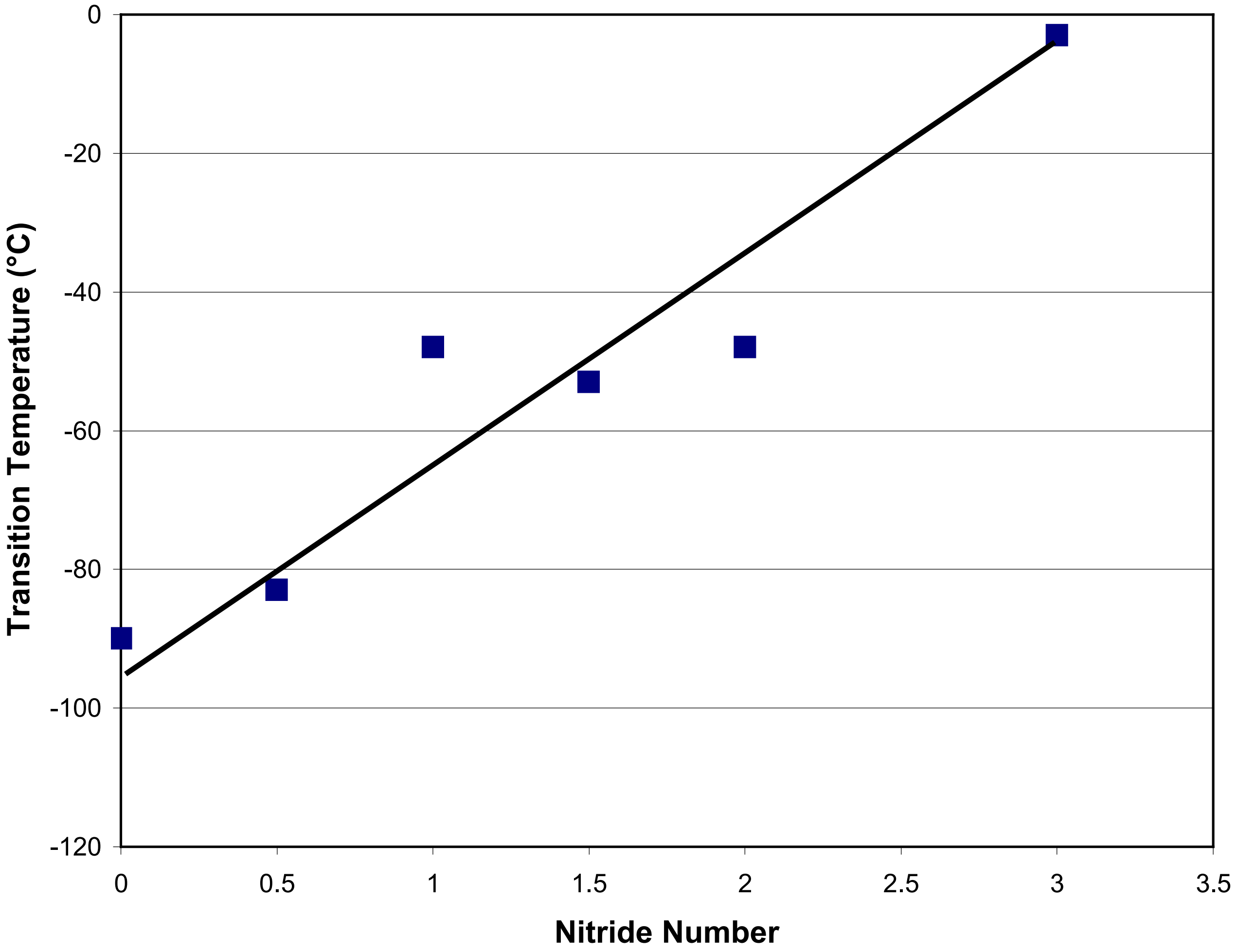
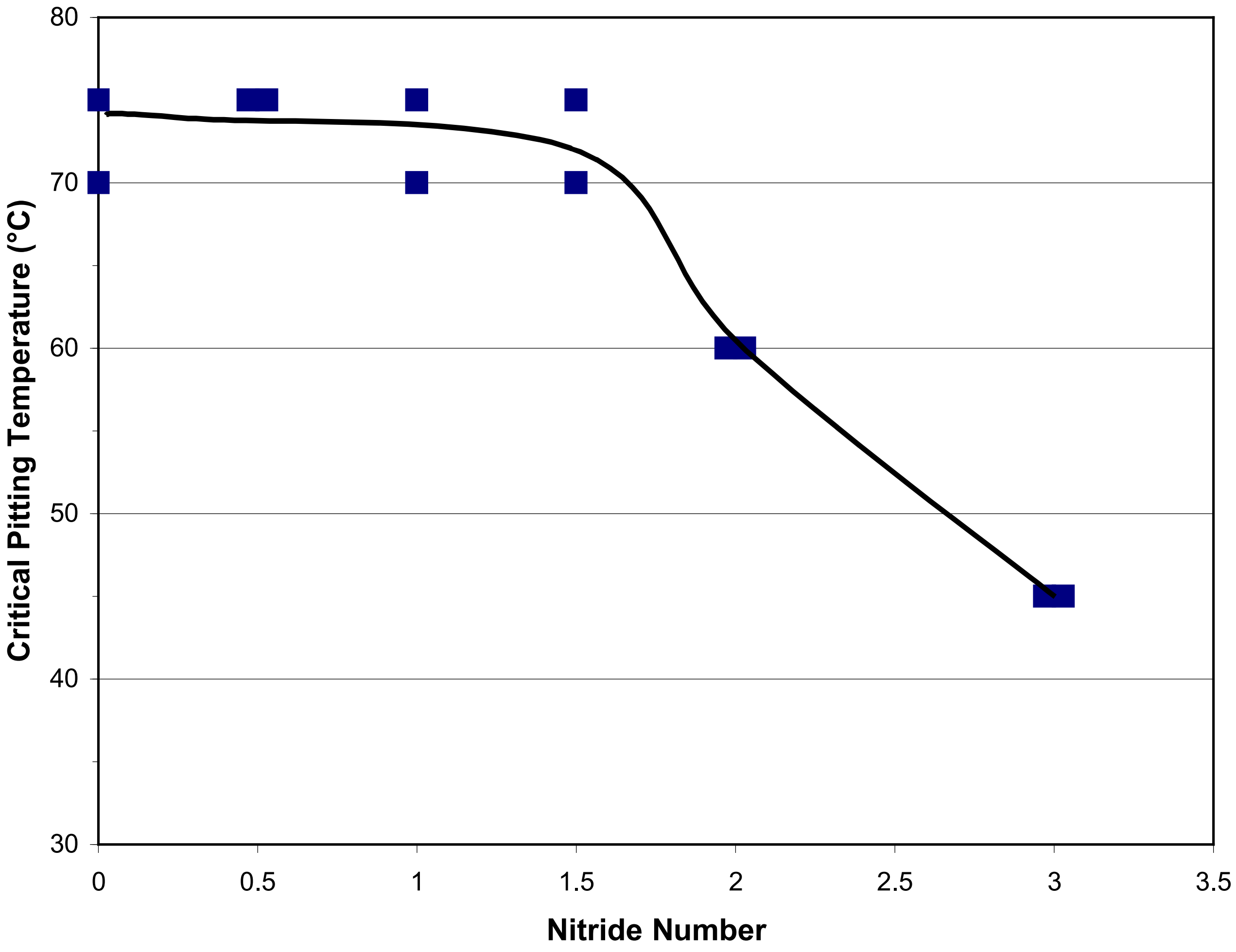


FIGURE 8 Critical pitting temperature in ferric chloride solution versus nitride number



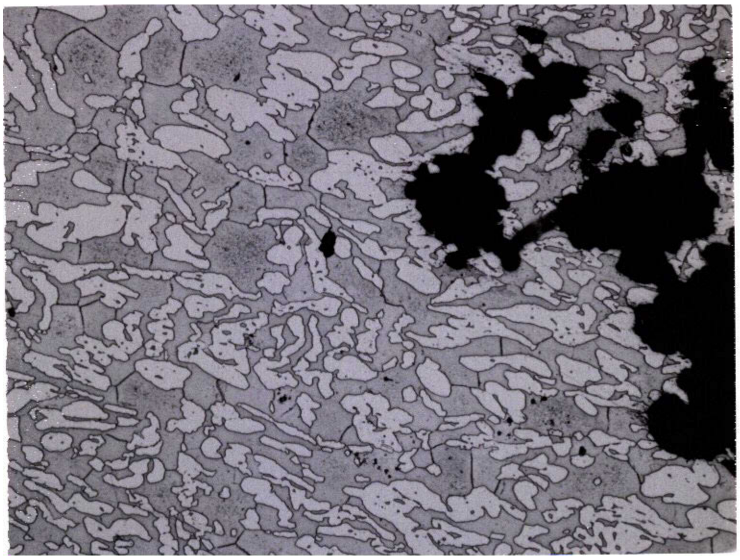


FIGURE 9 Microsection of ASTM G48 sample after test (Nitride number 3) [X100]

FIGURE 10 Critical pitting temperature in sea water versus nitride number

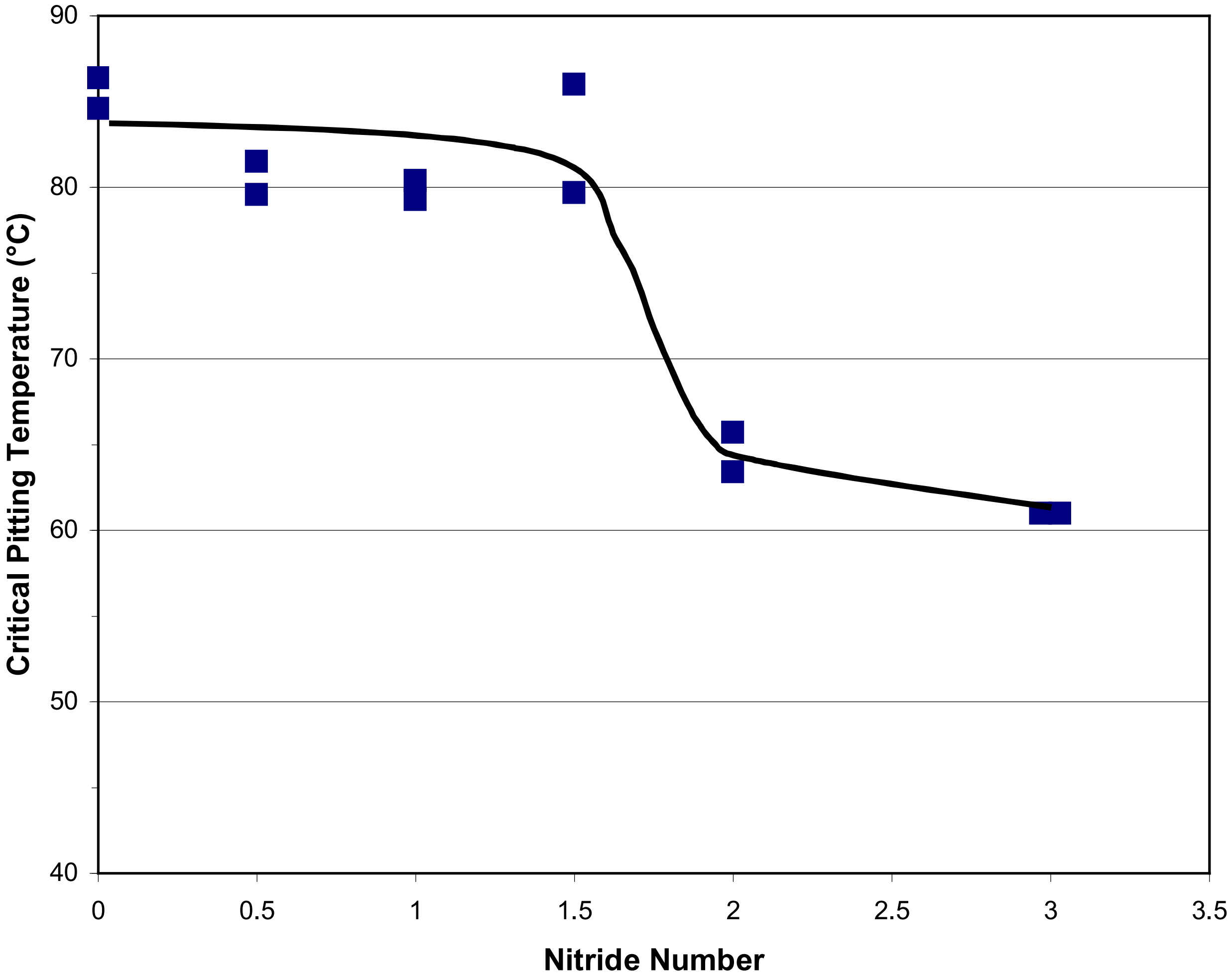


FIGURE 11 Stress versus time to failure under hydrogen charging

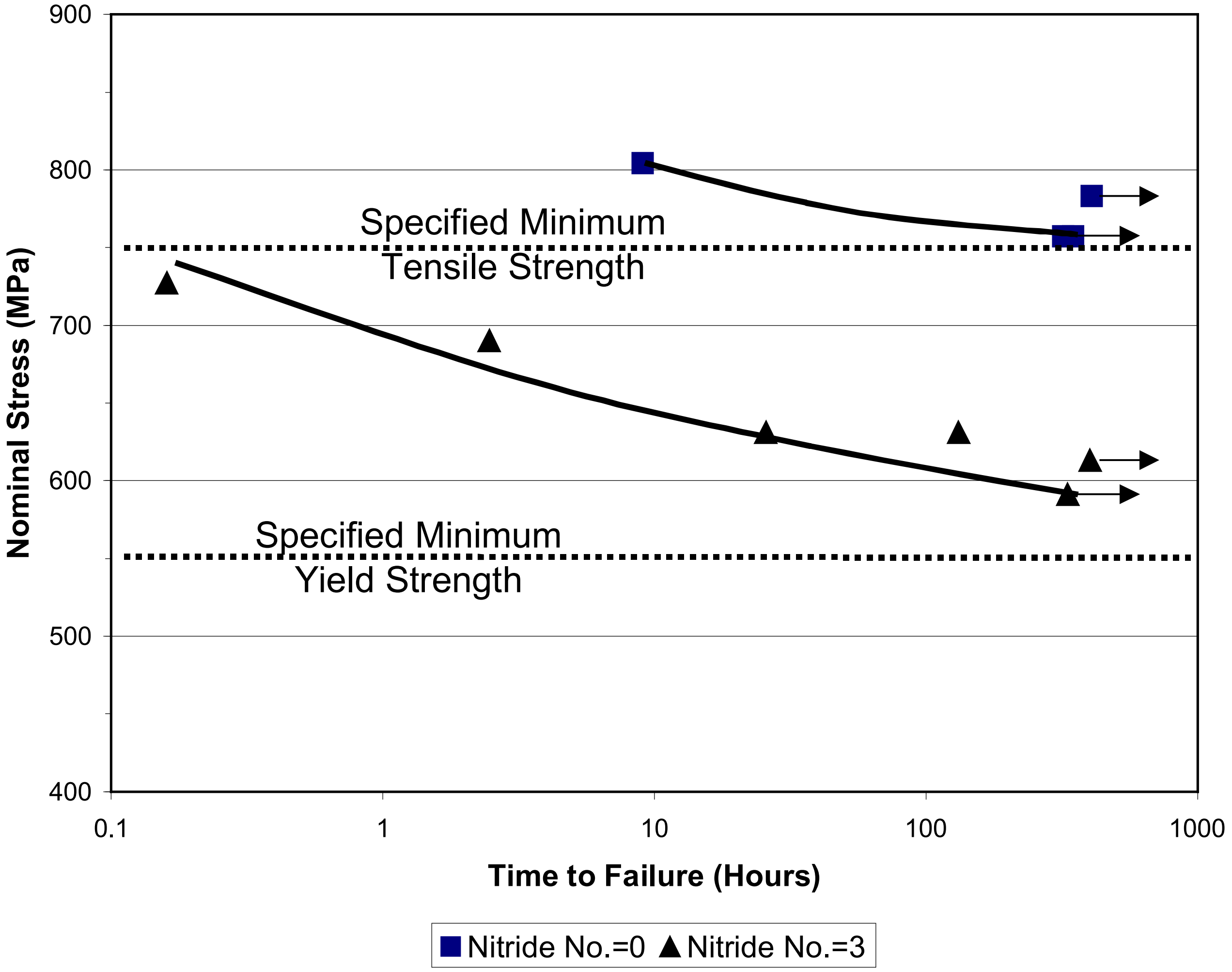


FIGURE 12 Plastic strain ratio under hydrogen charging versus nitride number

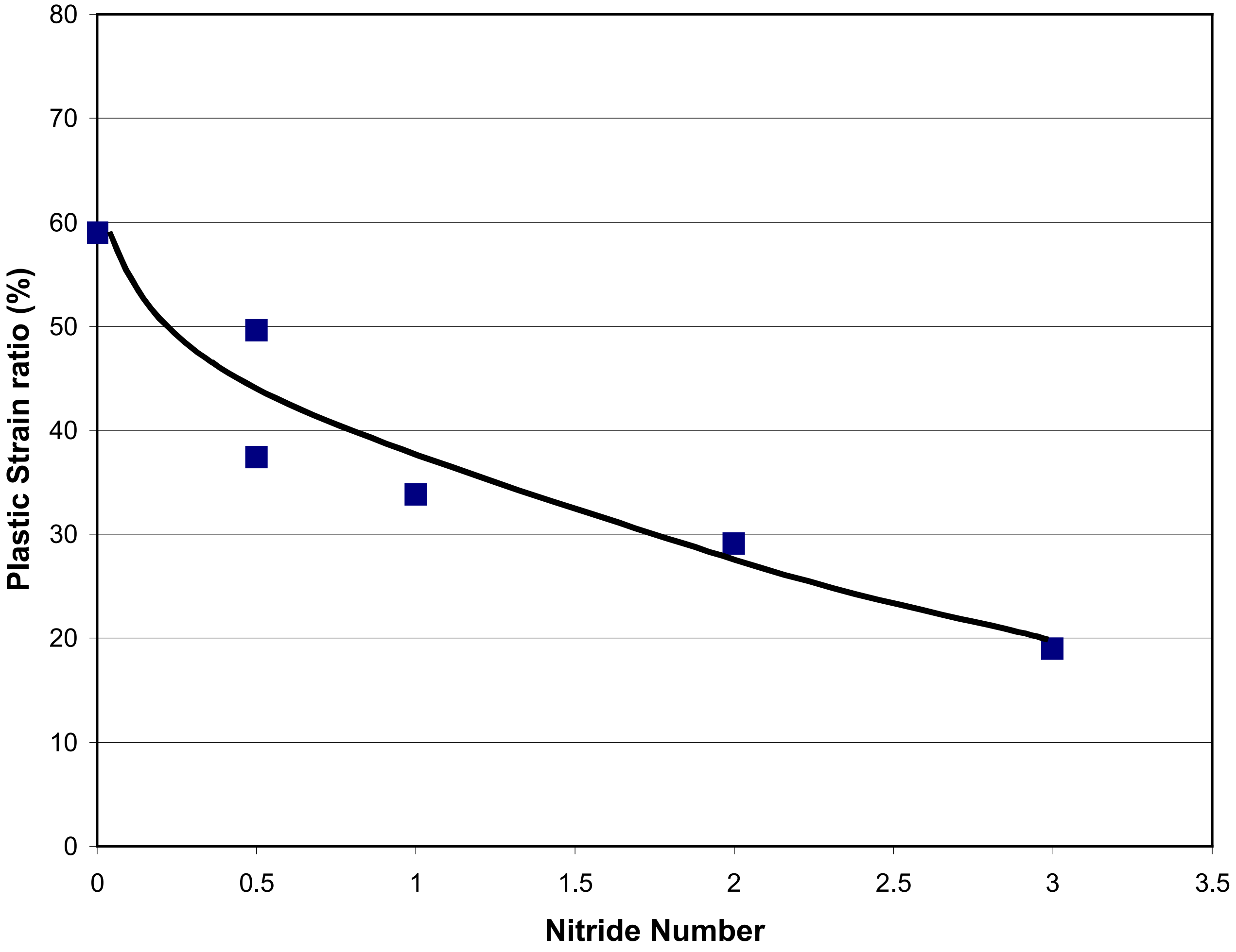
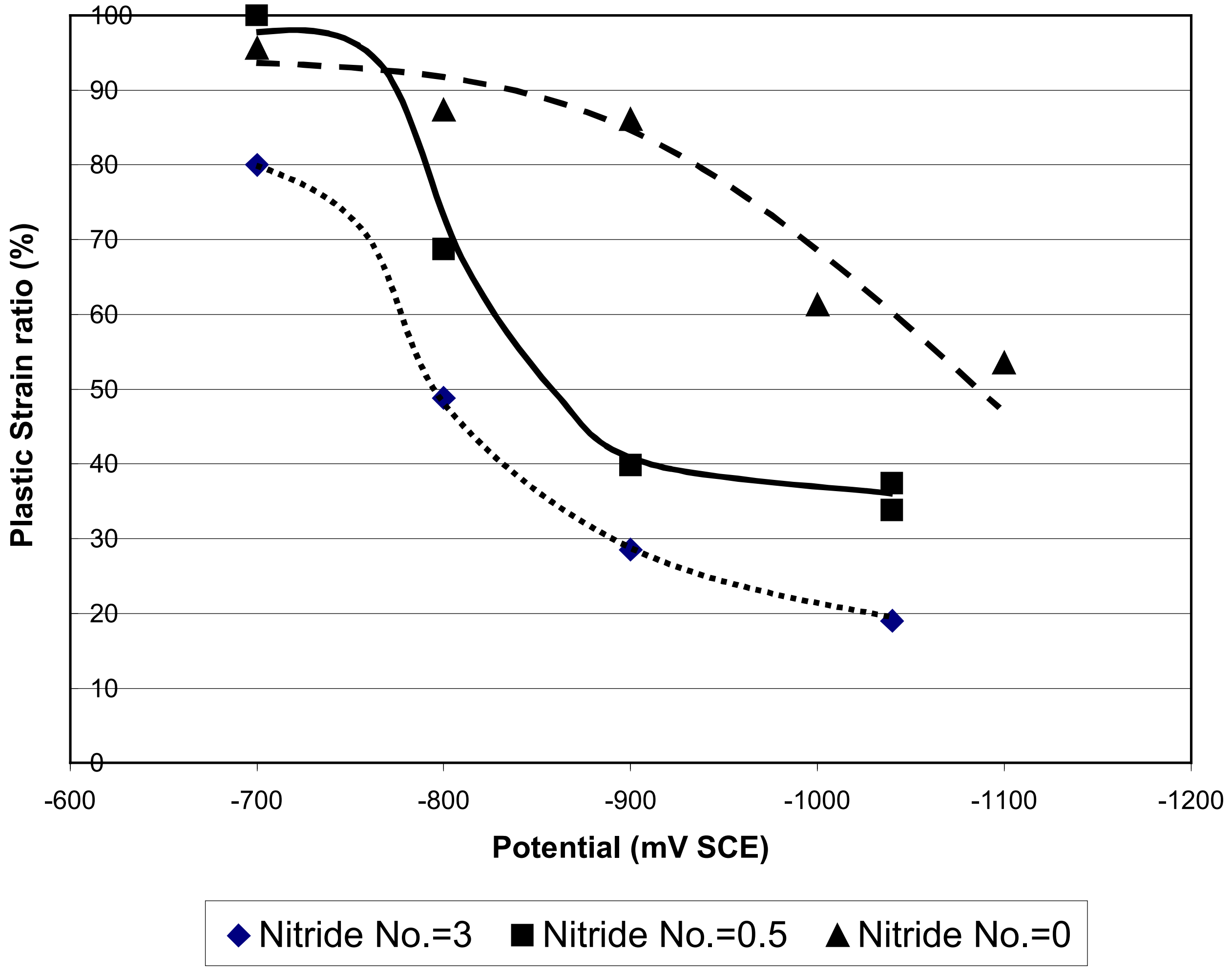


FIGURE 13 Plastic strain ratio under hydrogen charging versus potential



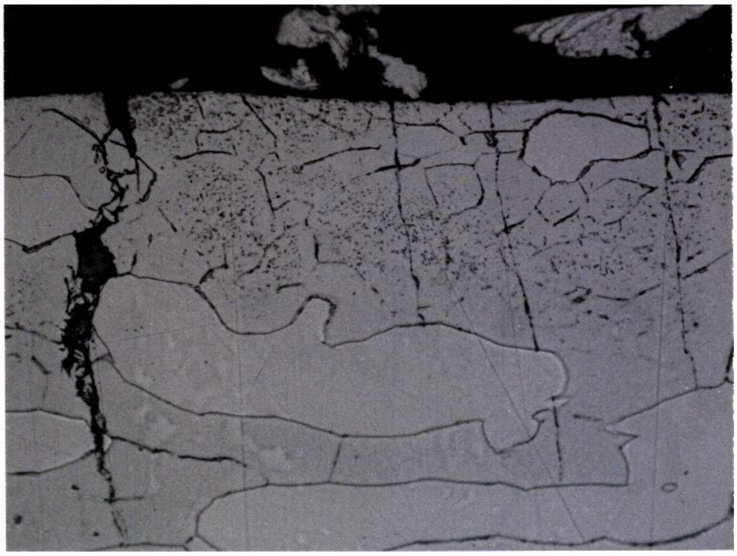


FIGURE 14 Microsection of OC9943 after slow strain rate testing under hydrogen charging (nitride number 3) [X500]