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Hardness And Nickel-Based Alloys

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ABSTRACT

The issue of hardness testing and nickel base alloys has been a contentious topic in the oil and gas community. In this paper we present the uses of hardness testing with respect to nickel base alloys with examples of limitations and applications. As hardness has historically been seen as a factor for predicting environmental cracking resistance of nickel base alloys, a literature assessment of slow strain rate data is presented examining the potential for a relationship between the hardness and the alloy's resistance to environmental cracking. More recent studies tried to access the mechanisms behind the environmental cracking issue and their results are reviewed.

The paper concludes that there is no reliable relationship between hardness and environmental cracking resistance on nickel base alloys and proposes some actions as a result.

Key words: hardness, nickel base alloys, UNS(1) N07718, HISC, environmental cracking, SSRT

INTRODUCTION

This paper addresses the relationship between hardness and environmental cracking resistance in nickel base alloys. The work here builds on the presentation made to AMPP's SC08 Fall 2021 meeting on October 19th.[1]

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(1) Unified Numbering System for Metals and Alloys (UNS). UNS numbers are listed in Metals & Alloys in the Unified Numbering System, 10th ed. (Warrendale, PA: SAE International and West Conshohocken, PA: ASTM International)

We use hardness as a measure of acceptance for a number of material property attributes. Some examples include using hardness values to indicate strength level, stiffness, resistance to scratching or abrasion, wear resistance, weldability, heat treat condition and resistance to environmental cracking. The accuracy or value of hardness to predict or estimate these characteristics lies in the relationship between hardness and the characteristic of interest. Hardness is the resistance to indentation. For some of these predictive characteristics, the relationship is pretty straightforward, for others, much more tenuous.

Hardness testing is universally used as a basic quality assessment tool for materials because of its simplicity of use, speed at which results are obtained and relatively low cost to apply. In addition to the relevance of the test method to assess the attribute of interest, we need to also be cognizant of the test details and the potential sources of variability and error. This is briefly discussed below.

For the purposes of this study, we are narrowing our field of interest to the relationship between hardness and the susceptibility to environmental cracking in the presence of hydrogen sulfide or dissolved hydrogen. Though we briefly comment on the relationship between hardness and environmental cracking for a variety of metallic materials, we further limit our more in-depth analysis to nickel base alloys.

To examine the relevance of hardness with respect to environmental cracking, we searched the literature for data that has both a measurement of hardness as well as a measure of resistance to environmental cracking in nickel base alloys. The first issue encountered was that there was a large quantity of references that had tests of resistance but the data was in the form of passing a set of criteria without a number or measure with which comparisons can be made. To overcome this, we centered our efforts on locating test data that provides a number or measure of cracking resistance. The slow strain rate test method (SSRT) where the ductility of samples tested in aggressive medium are compared to the ones of samples tested in inert medium provided the data used herein.

The second issue was data from different test methodologies; from autoclave testing with hydrogen sulfide containing environments to tests under conditions of hydrogen charging to assess resistance to hydrogen induced stress cracking (HISC). We elected to present all of the data.

The third issue was the presence of good cracking resistance data but the hardness measurements were not in HRC units or not present at all. The cold worked solid solution nickel-based alloys rarely listed hardness values of any kind. When the mechanical property data was presented but with no hardness values, we estimated the hardness value in Hardness Rockwell C (HRC) units from the tensile data. We recognize that this provides only rough values and we note when hardness values are estimated and provide the relationship between tensile and hardness values.

Lastly, we combined analyses of factors that influence environmental cracking resistance in nickel base alloys from the literature with the research data obtained from internal investigations. These analyses form the basis of our conclusions.

HARDNESS TESTING

Hardness testing is universal as a quick check to measure compliance with specified requirements that in the vast majority of cases includes hardness criteria. In the Oil & Gas industry hardness has been used as a go no-go with respect with resistance to environmental cracking in the presence of hydrogen sulfide in carbon & low alloy steels since the early 1950's. A number of papers were published in the Corrosion Journal in 1952 such as Bowers et al[2] that discuss hardness and cracking susceptibility in H_2S containing environments. In addition to the presence of other, perhaps more relevant variables than hardness, there are inherent factors in hardness testing that lead to errors and uncertainty in the resultant values.

Challenges with Measurement

Hardness measurements are simple and easy as noted but this does not imply that there are not some substantial sources in testing that leads to errors. A good discussion on hardness testing can be found in ASM Handbook Vol. 8[3]. Some sources of measurement errors are discussed below but more in-depth treatments of the subject are presented by Herring[4] and McGhee[5].

In addition to human error, there are potential measurement errors due to the test equipment and fixturing for the hardness test. For example, indenters can wear or exhibit spalling and testing machines have mechanical parts that can wear. Fixturing is extremely important because the indenter and test surface need to be perpendicular to each other and must be properly supported such that there is no movement during the test. The surface tested needs to be parallel with the anvil. Grit & contaminants in the test machine, test piece or fixtures will introduce testing errors.

Sample surface preparation is another critical area that can introduce huge sources of error. Rough or curved surfaces are sources of error. Also, the potential for cold working the surface tested is a potentially huge source of measurement error. For metals such as austenitic stainless steels and nickel base alloys, this can be more of an issue because of the greater strain hardening effects associated with these alloys. For example, in UNS S31603, even small amounts of cold work have been shown to result in hardness increases from about 84 Rockwell B in the solution annealed condition to about 23 HRC[6]. In UNS N07718, one study[7] demonstrated that under specific conditions with about 15% cold work, the hardness in the solution annealed and aged condition at about 44 HRC increased to about 48 HRC. As a further example, Sonmez and Demir[8] demonstrated that in a mild steel the Vickers hardness increases from about 160 to about 200 with only 0.25% strain.

Temperature is known to be a factor in variations in measured hardness. A measurable change in hardness value was demonstrated even with a temperature variation of 10°C.[9]

Variability in hardness results

With good hardness test practice, we can mitigate the majority of the sources of testing error noted previously. What remains is the sensitivity of prepared test surfaces to cold working with the resultant artificial increase in hardness and the fact that hardness test machines have a range from calibration data. Each machine, with everything else being equal, will exhibit a hardness result that could be different from other machines though each machine under evaluation demonstrates that it is in calibration. The calibration hardness test block itself has a hardness range with the average being the stated value of the test block.

In general, the harder the calibration test block is, the narrower the range with lower standard deviations. NIST(2) performed a study where they evaluated hardness test blocks from 6 manufacturers that were roughly about 25 HRC, 45 HRC and 65 HRC[10]. In Table 1, the hardness measurements taken by NIST and the manufacturer are reported for the 45 HRC range test blocks with the standard deviation and reproducibility. As demonstrated by NIST in Table 1, the hardness reproduceability measured on a hardness calibration block varies by 0.4 HRC to 1.2 HRC. The average hardness reproduceability of the six blocks tested by NIST is 0.6 HRC.

We can get an idea of the potential variability of hardness on machined surfaces due to machine and hardness calibration test block coupled with surface hardening due to machining operations. For a 40.0 HRC test block, the reproduceability is no better than +/- 0.3 HRC. For a modest + 2 HRC increase due to machining (cold work), it would not be unreasonable to measure hardness values that would be 0.3 HRC lower or 2.3 HRC higher from the actual bulk hardness value. Using 40.0 HRC, this could result in a range of 39.7 – 42.3 HRC.[2]

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(2) National Institute of Standards and Technology (NIST), Gaithersburg, Maryland, 20899

Table 1
Hardness Test Block Assessment Performed by NIST[9]

Manufacturer	1	2	3	4	5	6
Hardness values by NIST	46.5	44.5	45.0	44.8	44.0	44.0
	46.3	44.4	43.8	45.0	44.0	43.8
	45.9	44.5	44.6	45.2	43.8	43.9
	46.2	44.2	44.6	45.0	43.5	43.8
	46.0	44.0	44.6	45.0	43.8	43.6
average	46.2	44.2	44.4	45.0	43.8	43.8
standard deviation	0.24	0.22	0.54	0.14	0.20	0.15
reproducibility	0.6	0.5	1.2	0.4	0.5	0.4
Hardness values by manufacturer	47.2	44.4	44.1	46.0	44.5	44.3
	47.2	45.2	44.0	46.0	44.7	44.4
	47.2	44.4	44.2	46.1	44.4	44.4
	46.9	44.8	44.1	46.0	44.4	44.4
	47.5	44.6	44.0	46.0	44.7	44.4
average	47.2	44.7	44.1	46.0	44.5	44.4
standard deviation	0.21	0.33	0.08	0.04	0.15	0.04
reproducibility	0.6	0.8	0.2	0.1	0.3	0.1

HARDNESS AND SUSCEPTIBILITY TO ENVIRONMENTAL CRACKING

We have used hardness as one of measures of resistance to environmental cracking since at least the early 1950's. Early researches[11] into H_2S associated cracking demonstrated a correlation between hardness and probability of cracking in service. For example, Hudgins et al[12] published a comprehensive study evaluating the relationship of a variety of heat treatments with the resultant hardness values with time to failure as a function of H_2S concentration and hardness. However, even as hardness was being used as a criterion for steels, Bowers[2] noted in his summary "*Hardness as ordinarily measured is not a precise criterion in determining the susceptibility of steels to the failure process. Other mechanical properties are similarly deficient*." Even considering carbon and low alloy steels, it was recognized that microstructure played a critical role in resistance to cracking in H_2S (see, for example, Bowers[2] and Snape[13]).

Hardness as a measure of cracking resistance was also demonstrated on martensitic stainless steels[14] and 22 HRC, though a rough indicator of performance, was incorporated as the benchmark requirement in the historic NACE MR0175(3)materials requirement document from its earliest days[15]. Although the hardness acceptance level changed for other materials in the standard, hardness was still present as acceptance criteria. Nevertheless, dealing primarily with carbon and alloy steels, Caldwell[16] et al provides a good historical treatment of hardness and environmental cracking. The original concept of hardness

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(3) NACE MR0175, "Sulfide Stress Corrosion Cracking Resistant Materials for Oil Field Equipment"

versus performance with respect to susceptibility to environmental cracking proved valid when the tensile strength level as measured by hardness has a strong correlation with susceptibility.

The relevance of hardness as a predictor of environmental cracking resistance lies with the strength of the relationship between tensile strength and cracking resistance amongst the other variables that effect resistance.

For many corrosion resistant alloys such as duplex, highly alloyed austenitic stainless steels and nickel base alloys, we believed that the hardness was not a good indicator of resistance to environmental cracking. This has not been universally recognized but we have examples in our current NACE MR0175/ISO 15156[17] where solid solution nickel base alloys and duplex stainless steels have no hardness limits.

Source of data

As noted previously, to compare the relevance of hardness with resistance (or susceptibility) to environmental cracking, we need to have a recognized measure of hardness and a recognized measure of susceptibility to cracking. The hardness component was fairly straightforward but there was one reference that provided tensile data but not hardness data[18], here we estimated the hardness from the tensile data. The issue with a measure of cracking susceptibility was more difficult in that most of the work published was used to demonstrate applicability where tests were conducted and no evidence of cracking was observed. The data that was available for nickel base alloys was from the slow strain rate test (SSRT).

In searching through the literature data, we found that the NACE (now AMPP) Corrosion Conferences proved to be the source for most of the data that was collected. The sources of data with reference numbers, the materials that had data used here and some measure as to how the SSRT was conducted used in this paper were summarized and presented in Table 2 for the precipitation hardening nickel alloys.

Table 2
Precipitation Hardening Nickel Alloy Data Source

Ref.	abbreviated source	SSRT environment	Material(s) tested - UNS numbers
18	C2019 Paper 12948	25% NaCl, 400 psi H_2S, 800 psi CO_2, 149°C	N07718 bar stock
19	C2003 Paper 3126	varied NaCl, H_2S, CO_2 & temperatures	N07718, N07725, N09925
20	C2014 Paper 3948	0.5M H_2SO_4 @ 40°C 5 mA/cm^2 charging	N07718, N07716, N09925, N09935
21	C2014 Paper 4248	-1100 mVSCE for 48 hours	N07718, N07716, N09925, N09945
22	C2015 Paper 6053	3.5% NaCl, -1100 mV SCE	N07718, N07716, N09925
23	C2017 Paper 9068	0.5M H_2SO_4 @ 40°C 5 mA/cm^2 charging	N07718 bar stock
24	C2019 Paper 13161	-1100 mVSCE for 48 hours	N07718, N09945, N09946
25	C2019 Paper 13455	0.5M H_2SO_4 @ 40°C 5 mA/cm^2 charging	N07718, N07725, N09925, N09935, N09945, N09946, N07716
26	C2021 Paper 16821	0.5M H_2SO_4 5 mA/cm^2 charging	N07718
27	C2005 Paper 5103	7.5% NaCl, 3.5 MPa H_2S, 3.5 MPa CO_2, 121°C	N07718, N09925
28	C2012 Paper 1393	25% NaCl, 3.5 MPa H_2S, 3.5 MPa CO_2, 205°C	N07718, N09945
29	C2015 Paper 5911	0.5M H_2SO_4 @ 40°C 5 mA/cm^2 charging	N07718, N09945

30	C2018 Paper 11478	25% NaCl, 2.8 MPa H_2S, 5.5 MPa CO_2, 150°C	N07718 bar stock
31	C2021 Paper 16673	0.5M H_2SO_4 5 mA/cm^2 charging	N07716, N09955
32	C2015 Paper 5502	cathodic polarization to ESS-I-130	N07718, N09955
33	C2022 Paper 17966	0.5M H_2SO_4 5 mA/cm^2 charging	N07718, N07725, N09945, N09946

UNS N07718 Hardness – SSRT data

The advantage with the alloy that we have been most interested in, UNS N07718, is that the alloy is available in different grades that are produced using more than one heat treat procedure and there is a range of strength levels. This results in a fairly wide range of hardness values. Limiting the data to UNS N07718 data, the relationship between hardness and the ratio of elongation values obtained from test environment data with a control in inert medium is presented in Figure 1. The UNS N07718 data with the relationship between hardness and the reduction of area ratios is presented in Figure 2.

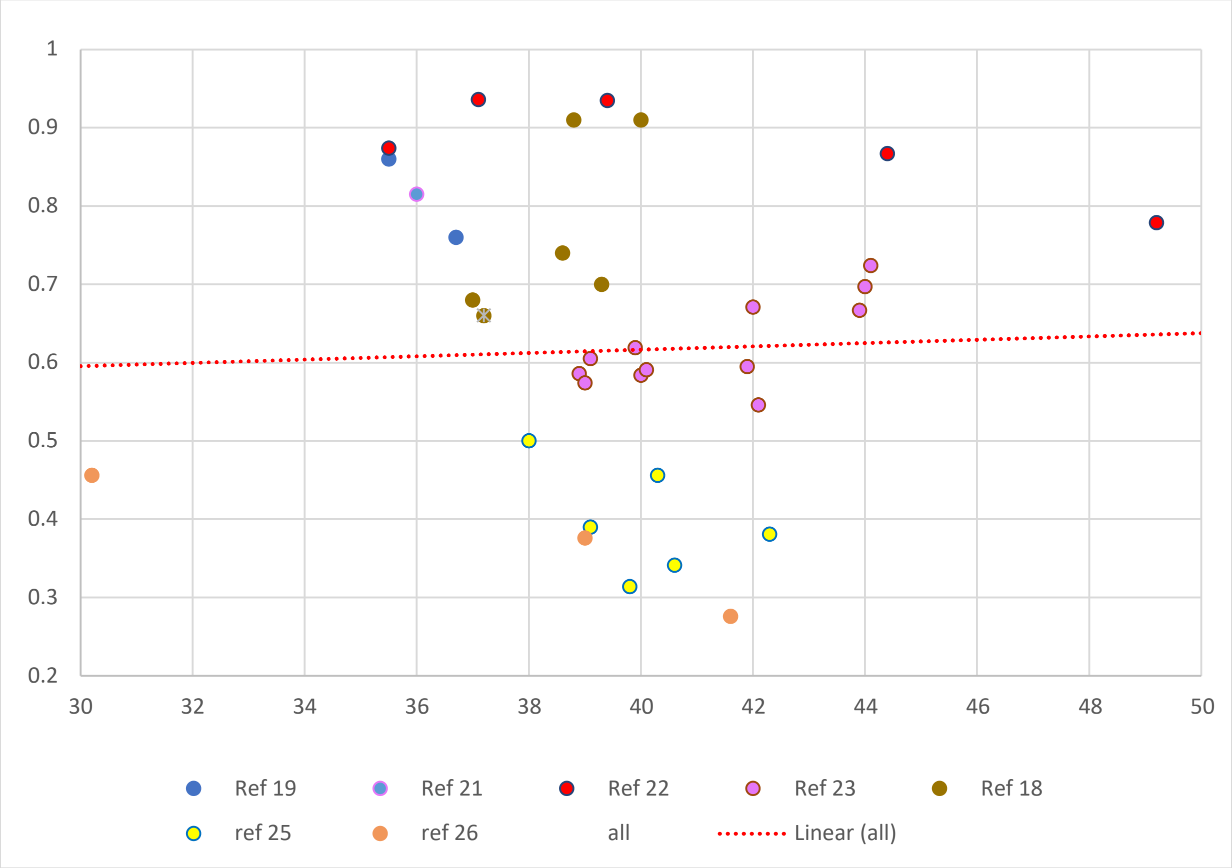

Figure 1: SSRT Elongation Ratios versus Hardness for UNS N07718

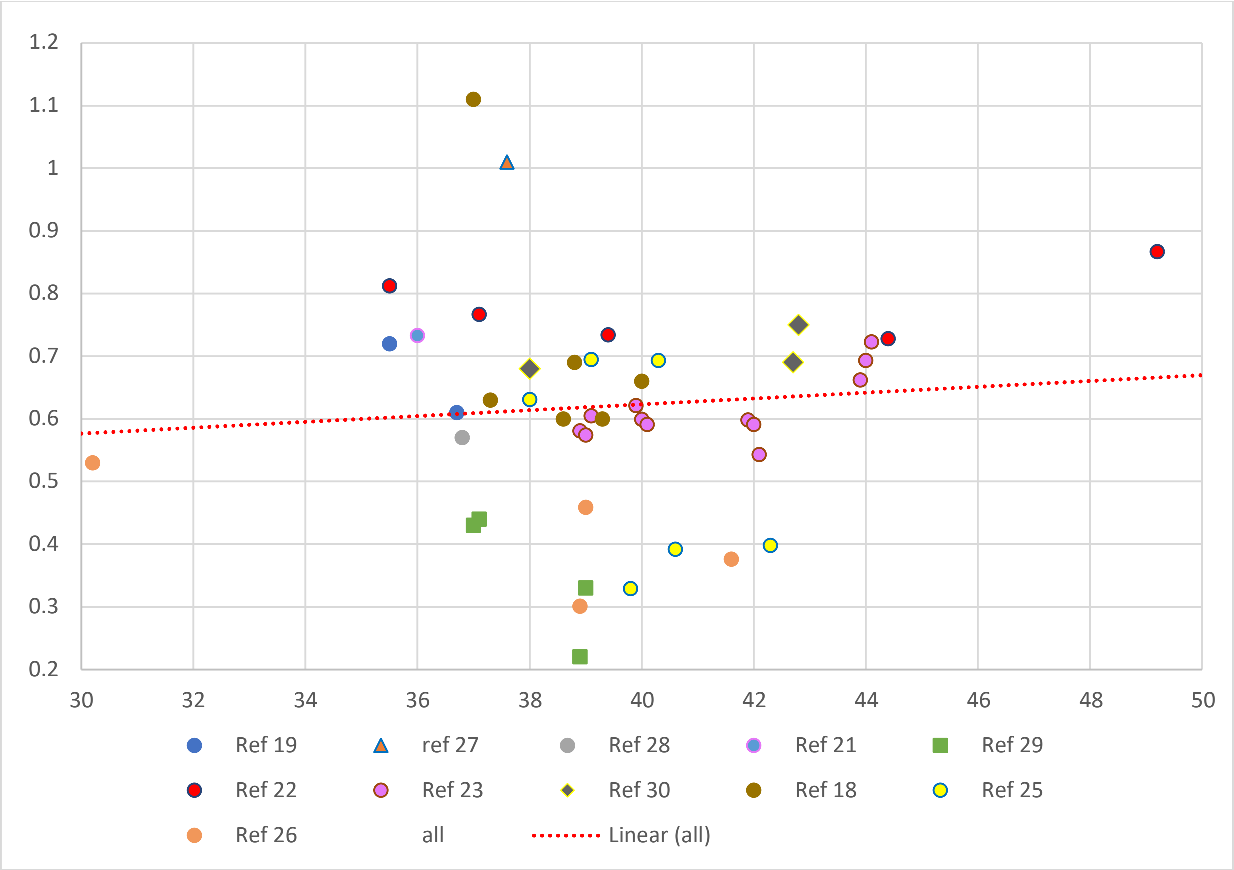

Figure 2: SSRT ROA Ratios versus Hardness for UNS N07718

From Figures 1 and 2 we find a lot of scatter but the trend lines for both the elongation and reduction of area ratios has a slight upward trend. For UNS N07718, the data does not show a clear trend between hardness and the environmental cracking susceptibility as measured by the SSRT.

In opening the scope to include all precipitation hardening nickel base alloys, we find similar relationships between SSRT ductility ratios with hardness. The relationship between hardness and the ratio of elongation values obtained from test environment data with a control in inert medium for all of the precipitation hardening alloys is presented in Figure 3. Similarly, the data with the relationship between hardness and the reduction of area ratios is presented in Figure 4.

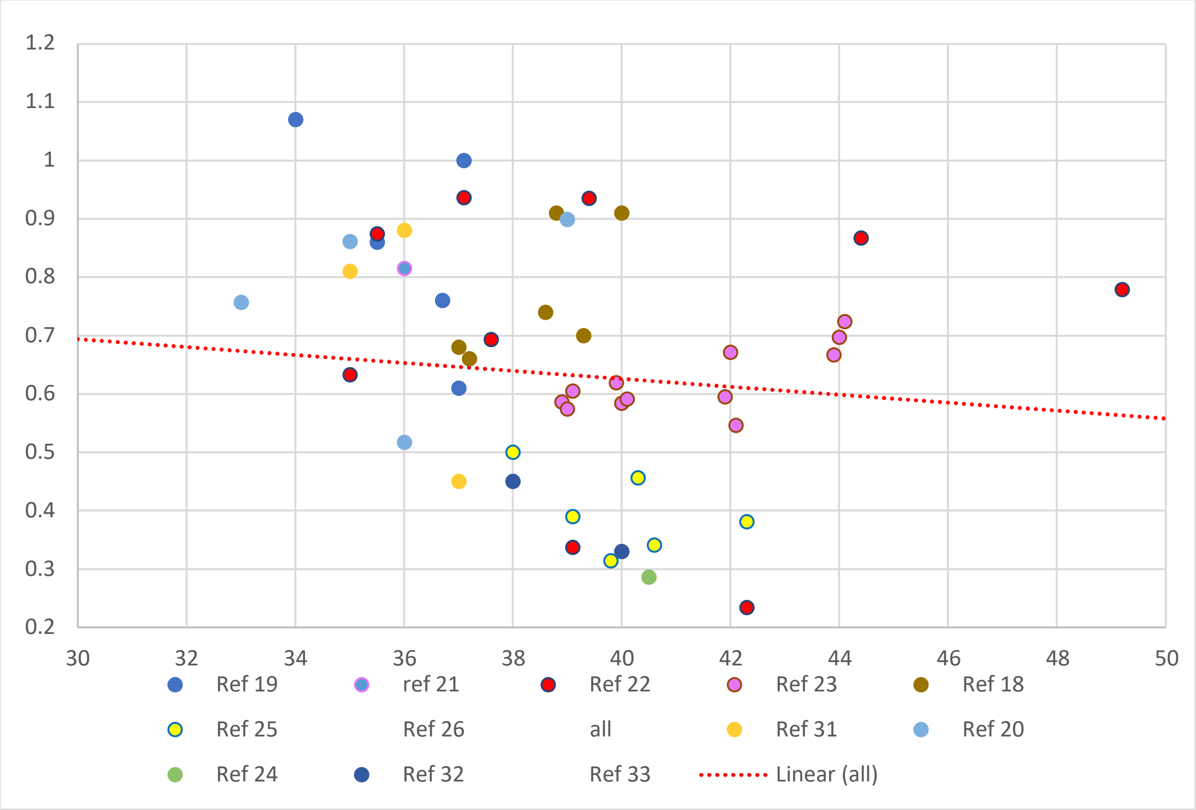

Figure 3: SSRT Elongation Ratios versus Hardness for all Precipitation Hardening Nickel Alloys

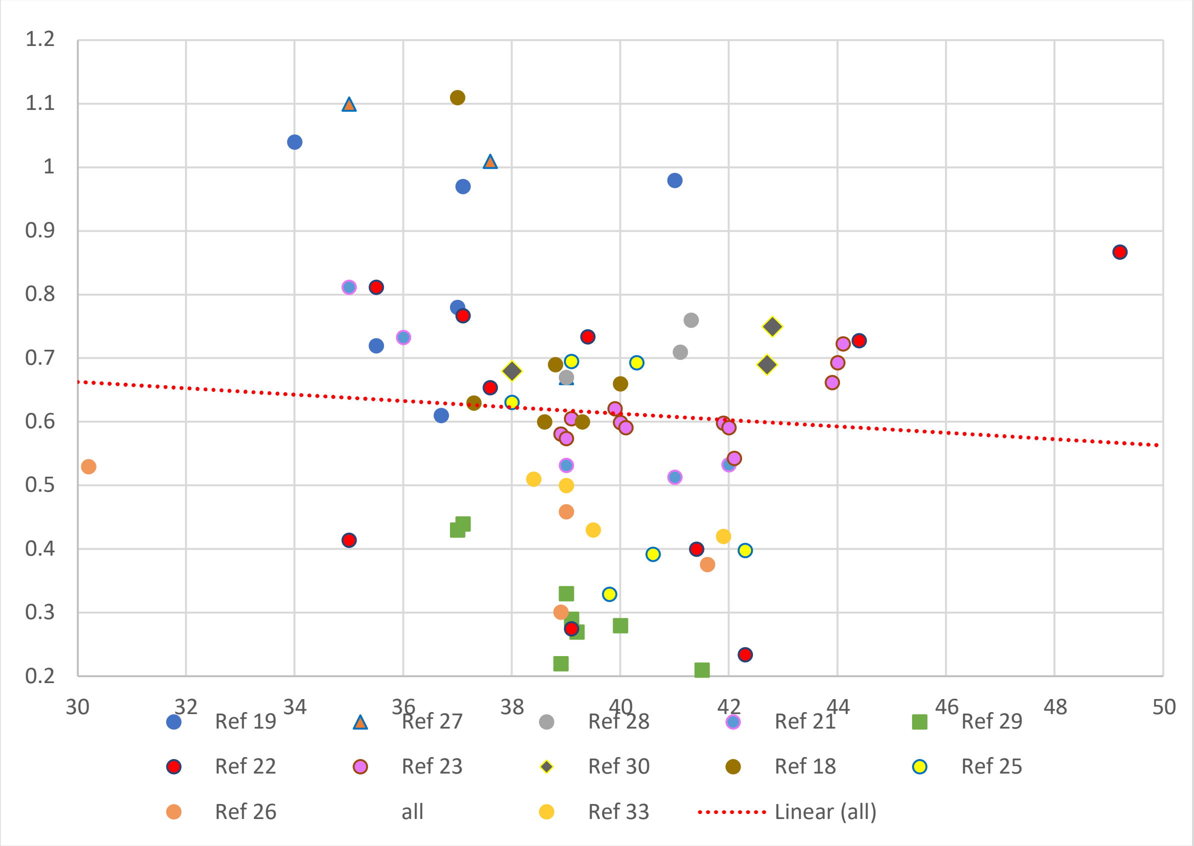

Figure 4: SSRT ROA ratios versus Hardness for all Precipitation Hardening Nickel Alloys

When considering all precipitation hardening nickel alloys, the SSRT ratio versus hardness trend lines rake slightly downward with scatter. Again, the SSRT data demonstrates the lack of a clear relationship between hardness and environmental cracking resistance as measured by SSRT.

The correlation between hardness and susceptibility to environmental cracking for solid solution nickel alloys proved to be more difficult to document because only a few references present hardness data and, when present, usually as typical values. On the plus side, there are a few references that contain SSRT data for these alloys; these are listed in Table 3.

Table 3
Solid Solution Nickel Alloy Data Source

Ref.	abbreviated source	SSRT environment	Material(s) tested - UNS numbers
34	C1997 Paper 25	25% NaCl, 0.2 MPa H_2S, 5.5 MPa CO_2, 177°C	N08535
35	C2000 Paper 00149	10% NaCl, 0.69 MPa H_2S, 2.76 MPa CO_2, 204°C	N08028, N08825
36	C2001 Paper 01004	25% NaCl, 7 bar H_2S, O.5% HAc, 177°C	N08031
37	C2012 Paper 01684	25% NaCl, 0.7 bar H_2S, O.5% HAc, 150°C	29Ni-25Cr-3Cu-0Mo
38	C2022 Paper 17960	151 & 280 kppm NaCl, 100 & 300 psi H_2S, 0 & 180 psi CO_2, 149°C	N08028, N08825
39	C2000 Paper 0149	25% NaCl, 300 psi H_2S, 1000 psi CO_2, 218°C	N06985

Most of the data from the papers listed in Table 2 have SSRT elongation and reduction ratios that are at 0.9 and above. The materials tested were in the cold worked condition. The yield strengths listed ranged from a low of about 120 ksi (827 MPa) to a high of almost 150 ksi (1034 MPa). The lowest reported hardness was 28 HRC and the highest was 35 HRC.

The take-aways that we can glean from the limited data from the solid solution nickel alloys are (1) the hardness does not appear to be relevant and (2) the overall resistance to environmental cracking is better than the precipitation hardened alloys.

NICKEL ALLOY VARIABLES THAT CORRESPOND TO ENVIRONMENTAL CRACKING RESISTANCE

In the last few years, the published data related to the variables that correspond to environmental cracking resistance has increased and the focus has turned to the microstructural particularities that may have a role in the environmental cracking resistance. The role of microstructure was found to be most significant and Trillo et al[40] found that hardness was not a source of the variation in SSRT results; the testing performed showed large variations in hardness and there was no definitive correlation between elongation ratios and hardness. Duret-Thual et al[41] demonstrated that unacceptable microstructure was the most important variable and there did not appear to be a relationship between strength level and neither the elongation nor the elongation ratio under cathodic protection in SSRT tests.

UNS N07718 and other precipitation hardenable nickel alloys present complex structures containing several intermetallic phases, nitrides, carbides and carbonitrides. The intermetallics may constitute of gamma-prime and/or gamma-double-prime, and the stable eta and/or delta phases, and are composed mainly of nickel in combination with aluminum, titanium and niobium. The size and amount of these precipitates depend upon the heat treatment time and temperatures to which the alloy is submitted during its manufacturing.

Klöwer et al[23] demonstrated in 2017 through different age hardening heat treatments, producing material in different conditions, including non-conforming to the NACE MR0175 and API(4) Standard 6ACRA[42] requirements, that the intermetallic phases gamma-prime, gamma-double-prime and delta influenced the hydrogen diffusion in UNS N07718. They concluded that large amounts of delta phase increase the susceptibility of UNS N07718 to hydrogen embrittlement, but if the ageing was carried out within the temperature range of the API 6ACRA, and the microstructure was in conformance to the acceptable microstructures as available in Annex A, the effect of this phase was negligible. Different conditions having different gamma-prime and gamma-double-prime precipitation sizes have been evaluated and the finer precipitates were correlated to a less susceptibility to hydrogen embrittlement.

In 2018 Rosenberg et al[43] published all the data that was generated in the context of the NACE ballot 2017-04 to include the 150 ksi (1034 MPa) grade to NACE MR0175 and later to API 6ACRA. The three available grades with minimum 120, 140 and 150 ksi (827 MPa, 965 MPa and 1034 MPa respectively) with varying hardness levels were tested on SSRT and no relationship could be identified between hardness and the SSRT ductility ratios, although the hardness behavior showed to have a linear correlation to the yield strengths.

In 2019, Morana et al[44] published a summary of several post failure analyses. An UNS N07718 tubing hanger failed in one of its areas affected with more stress. Dense acicular delta phase was detected in the grain boundaries, showing an unacceptable microstructure according to references of Annex A. An UNS N07718 casing hanger also failed in its more stressed area, but no heavy Delta phase has been reported, although some degree of precipitation in the grain boundaries was present and could be linked to the failure. Additional UNS N07725 cross-over and UNS N07716 sub-surface safety valve component failures took place in the regions of the components subjected to higher stresses, but grain boundaries were clear from secondary phases and no metallurgical features deemed detrimental by the API 6ACRA were localized. Zhang et al[45, 46] propose a mechanism that foresees the formation of nanovoids along dislocation slip bands and specially at their intersections - when defects merge, it results in crack initiation.

More recently Botinha et al[47,48,49] demonstrated that the intermetallic precipitates gamma-prime and gamma-double-prime play an important role in the hydrogen embrittlement susceptibility of UNS N07718. Through ab-initio simulations and by varying the gamma-double-prime amount in UNS N07718 (by heat treatment or adaptation of chemical composition) they conclude that the hydrogen atoms interact with the interfaces of gamma-double-prime with the matrix of the alloy, reducing the strength needed for cracking. They concluded therefore, that higher amounts of gamma-double-prime in the alloy microstructure increases the susceptibility of the alloys to hydrogen induced cracking.

DISCUSSION

In Reference 18, the SSRT data is presented with known values of yield and tensile strength but no hardness values. For the data points from this reference we used the hardness estimates based upon the measured yield strength and the corresponding hardness from a trendline that results from known yield and hardness measurements from the other references in this paper. The data and trendline are presented in Figure 5.

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(4) American Petroleum Institute (API), 1229 L St. N.W., Washington, D.C. 20005-4070

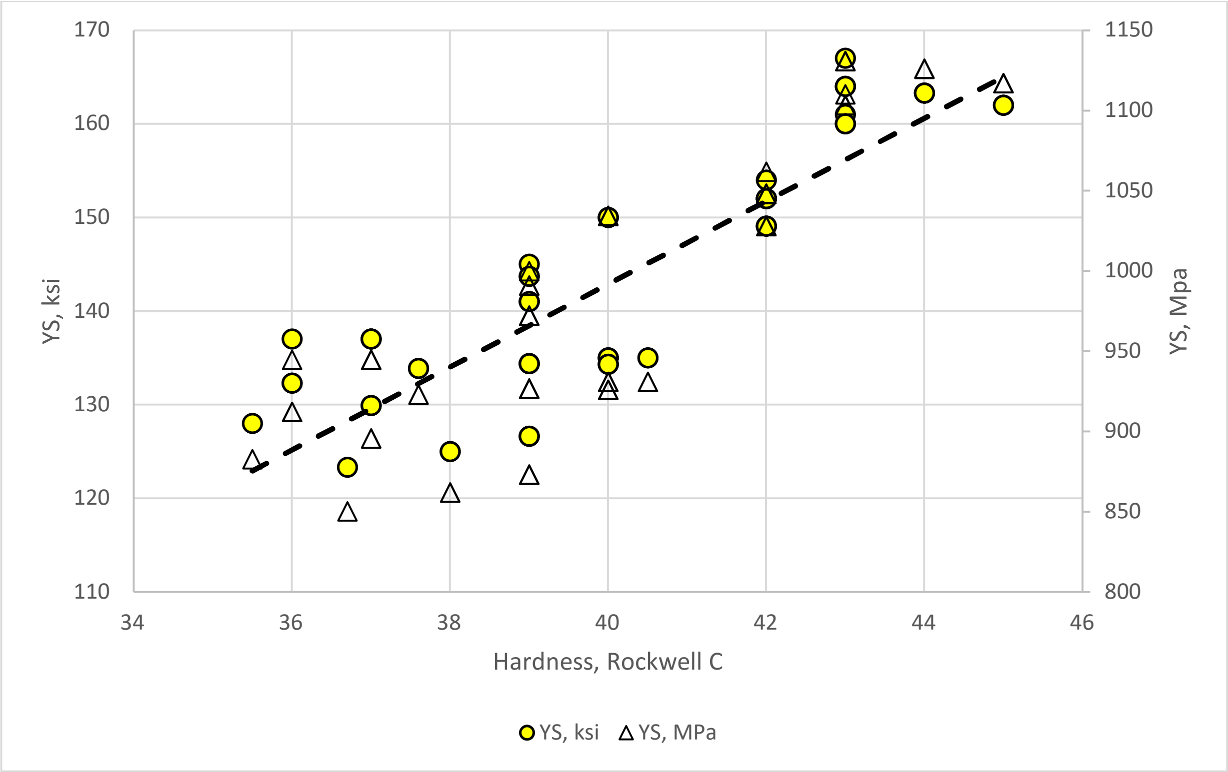

Figure 5: Relationship between yield strength and hardness with a linear dashed trendline

We know the value of hardness tests as a simple quality tool that gives us knowledge about the state of the material including heat treat condition. The question is what should we use hardness tests for in nickel alloys? From the data available in the literature and presented here, we could find no reasonable relationship between hardness and susceptibility to environmental cracking.

CONCLUSIONS

1) UNS N07718 (the largest data set by far) does not show a correlation between hardness and SSRT ductility ratios.
2) Data for all precipitation hardened nickel alloys does not show a correlation between hardness and SSRT ductility ratios.
3) The solid solution hardening nickel alloys do not show a correlation between hardness and SSRT ductility ratios.
4) As a group, the solid solution nickel alloys exhibited greater resistance to environmental cracking compared with the precipitation hardened nickel alloys as a group.
5) Microstructure is the overriding factor with respect to susceptibility to environmental cracking; the grain boundary condition and the presence of secondary phases.
6) Gamma-prime seems to be not harmful for the hydrogen induced cracking resistance of precipitation hardenable nickel base alloys, while gamma-double-prime seems to be deleterious.Higher ratios of gamma-prime to gamma-double-prime are preferential for the hydrogen induced cracking resistance.
7) We should use hardness testing for nickel base alloys as an indicator of heat treated condition for quality purposes and not as a specified limit for resistance to environmental cracking.

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