PROPERTIES AND PERFORMANCE OF A HIGH CHROMIUM NICKEL ALLOY FILLER METAL 52i

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ABSTRACT

The weld filler metal EN52i/E152i was developed by BMPC and Outokumpu VDM USA to possess both improved weldability relative to other high chromium nickel-based alloys and stress corrosion cracking resistance comparable to wrought Alloy 690. The following paper reports on an extensive testing program to determine the physical properties, mechanical properties, weldability, environmentally assisted cracking resistance, and phase stability of EN52i. Results show that EN52i has comparable physical properties, mechanical properties, weldability, and corrosion fatigue performance relative to the widely used but lower chromium filler metal EN82H. Low temperature crack propagation toughness is somewhat superior to EN82H but most notably, EN52i displays a significantly greater resistance to stress corrosion cracking in primary water. For example, under identical testing conditions in 680°F (360°C) high purity water, EN52i exhibits crack growth rates conservatively estimated ≥50X slower than EN82H. Similarly, in composite specimen ‘crack arrest’ type SCC tests, actively growing cracks were observed to dramatically decrease their crack growth rate in the first weld bead mixed with the higher chromium alloy EN52i. Notably, in all these tests, the SCC crack front was never fully engaged and did not propagate more than one grain in extent in tests that ran up to 2 years long. Isothermal heat treatments between 631-878°F (333-470°C) for times up to 3,000 hours indicate good metallurgical stability with no hardening indicative of long range ordering. Based on the results of this testing, EN52i is shown to possess a desirable combination of weldability, mechanical properties, phase stability, and resistance to environmentally assisted cracking.

Keywords: EN52i, nickel alloy weld metals, welding, weldability, stress corrosion cracking, low temperature crack propagation, corrosion fatigue.

1.0 BACKGROUND

The weld filler metal EN52i and the matching shielded metal arc welding (SMAW) electrode E-152 were designed by BMPC to be resistant to solid state ‘ductility dip’ cracking during welding and to greatly improve the stress corrosion resistance relative to lower chromium weld filler metals EN82H/E-182 [1] [2] [3]. This paper summarizes the results of an extensive experimental program on the weldability, physical properties, mechanical properties, environmentally assisted cracking resistance, and phase stability of this alloy. As will be shown, the alloy offers a desirable combination of properties that are suitable for use in critical applications like nuclear power systems.
2.0 EXPERIMENTAL PROCEDURE

For most properties, results from three heats of weld wire were obtained. Unless otherwise specified, the results are from multi-pass, automatic gas tungsten arc welds that were used to fabricate the test samples. Both v-groove and weld pad buildups were utilized for the testing. The composition of each heat of wire, as determined from an independent chemical analysis is given in Table I.

2.1 Physical and Thermal Properties

Physical and thermal property testing included work to determine the alloys’ density, mean coefficient of thermal expansion, specific heat, thermal conductivity, and thermal diffusivity. Replicate tests for each temperature and multiple heats of material were used to estimate the average response of the alloy. Additionally, for one heat (MLTS-2), the melting range was estimated via on-heating differential thermal analysis, conducted at a heating rate of 25°C/minute.

2.2 Elastic and Mechanical Properties

The Poisson’s ratio and Young’s modulus were determined by the impulse excitation of vibration technique as specified in ASTM E1876-09. For Poisson’s ratio, replicate tests in the L (parallel to the welding direction), S (short) and T (transverse) directions were assessed at room temperature. For the Young’s modulus testing, a fourth diagonal orientation was added that was ~45° off-axis relative to welding direction and testing was conducted between room temperature and 2200°F (1204°C). Tensile tests were performed on both gas-tungsten-arc (GTA) and shielded-metal-arc (SMA) welds in accordance with ASTM E8-14 in the as-welded and stress relieved conditions. Temperatures between 70°F (21°C) and 700°F (371°C) were investigated. Additionally, the effects of stress relief (1050°F/566°C for 30 hours) and orientation (L vs. T) were evaluated.

2.3 Weldability

The weldability of filler metal EN52 was assessed via several methods. These included transvarestraint testing, a dissimilar metal linear v-groove weld known to produce solid state ductility dip cracking in EN52, and a highly restrained linear narrow groove weld, known to produce solidification-type cracking in susceptible alloys. These dissimilar metal welds were performed with both A-GTAW and SMAW, while the narrow groove was welded with A-GTAW. For the narrow groove welds, two distinct regions (cold wire filler metal and hot-wire filler metal) were evaluated separately. Finally, the experience of industrial users in various applications of the weld filler metal was solicited as independent assessments of the filler metal.

2.4 Environmentally Assisted Cracking

The environmentally assisted cracking resistance of the alloy in hydrogenated water was assessed via low temperature crack propagation (LTCP) tests, corrosion fatigue crack growth rate tests, and stress corrosion cracking (SCC) tests. All testing was done with fracture-mechanics based methodology on compact tension samples in the T-S orientation. The LTCP tests utilized elastic-plastic fracture mechanics to determine the toughness in room temperature air (70°F / 21°C), 640°F (338°C) air, and in 130°F (54°C) water as a function of hydrogen concentration. Furthermore, the LTCP tests ‘preconditioned’ the samples with a 24 hour, 550°F (288°C), 60 sec H2 / kg H2O exposure to promote hydrogen pickup. For the SCC tests, two methodologies were used: (1) tests on monolithic samples of EN52i and (2) tests on composite welds where SCC is initiated in a susceptible material (E-182) and propagated toward EN52i. The composite samples were tested for 6, 12, 18, and 24 months in 680°F (360°C) high purity water at an initial stress intensity factor of 30 ksi√in (33 MPa√m).
3.0 RESULTS AND DISCUSSION

3.1 Physical Properties

As expected, the physical property tests displayed smooth, monotonic behavior over the temperature ranges studied. Table II summarizes the room temperature physical properties, along with the estimated melting range of the alloy. The effect of temperature on the density, mean coefficient of thermal expansion, specific heat, thermal conductivity, and thermal diffusivity is given in Figure 1.

3.2 Mechanical Properties

Poisson’s ratio ($\nu$) testing is summarized in Figure 2, which shows average values in the L, S, T, and diagonal ‘D’ orientations between 0.2 and 0.3. Given the scatter in the data, there is no clear effect of orientation and the average value of 0.26 is suggested as reasonable for the alloy. Figure 3a illustrates examples of crystallographic textures that have been observed in GTA welds of EN52i. As shown, significant variation in the type and degree of texture has been observed with relatively small changes in the welding parameters. For example, both $<100>$ pseudo-single crystal and $<100>$ fiber type texture have been observed in simple AGTA weld pad buildups [4]. As shown in Figure 3b, this crystallographic texture results in lower (~25-28 Msi) Young’s modulus when testing is conducted in the direction of the texture and higher modulus (~31 Msi) at 45° to the texture (i.e., in the $<110>$ direction).

The tensile data on EN52i and E-152i are summarized in Figure 4. Figure 4a presents data for EN52i as-welded (solid lines) and after a 1050°F / 30 hour stress-relief heat treatment (dashed lines). As shown, there is no adverse effect of the post weld heat treatment. Room temperature yield strengths are ~65 ksi, ultimate tensile strength ~95 ksi, and %Elongation and %RA $\geq$ 30%. Figure 4b compares the as-welded gas-tungsten-arc-weld tensile data to shielded-metal-arc welds. The data are essentially identical due, in part, to the unified composition between the weld wire and the shielded metal arc electrodes. Unlike many other nickel alloy filler metals (e.g., EN82H / E-182, EN52M / E-15M2, EN52 / E-152), filler metal 52i was designed so that a single composition is suitable for both the bare wire and the coated electrode. Lastly, note the relatively consistent properties with little effect of orientation. The one exception to this is the ‘T’ direction % elongations that were consistently lower than the ‘L’ tests, but were still highly ductile with average elongations $> 28%$.

3.3 Weldability

Results of transvarestraint testing are given in Figure 5a along with comparisons of the typical cracking at 5% applied strain in 5b. Solidification cracking resistance is compared via the maximum crack distance as a function of applied strain. In our Laboratory’s experience, maximum crack distance in the plateau region of the transvarestraint test is a good indicator of actual solidification cracking resistance in fusion welds since it essentially compares the solidification temperature range of each alloy [5]. As shown, the EN52i data lay near EN82H, with heat 187775 exhibiting somewhat higher propensity for hot cracking relative to heats MLTS-2 or 126373. This is likely due to the niobium concentration (2.58 wt.%) in this heat, a level that is now above the recommended Nb limit (2.2-2.4 wt.%) for the alloy.

The solid state cracking (i.e. ‘ductility dip’ cracking or DDC) resistance of several weld metals is assessed in Figure 6. This dissimilar weld metal joint (carbon steel to stainless steel) has been shown to produce and to discriminate the propensity to undergo DDC of several Ni-Cr weld metals [1, 2, 6]. The top plot summarizes data for automatic GTA welds and the bottom plot for manual SMA welds. For the GTA welds, other dissimilar metal side rail combinations were assessed, including 304L/A690, A600/EN82H, and 304L/308L. As shown, GTA welds with EN52i meet the target of $\leq$ 2 cracks in 8 metallographic cross sections and are far superior to EN52 which displayed ~15 cracks. For the SMA welds, EN52i also exhibited very good cracking resistance with far fewer cracks (5-10) relative to many other shielded metal electrodes (e.g. $> 25$ cracks for filler metals 152M, 152MSS, and 69HP).
A highly restrained narrow groove weld was also used to compare the weldability of several alloys [2, 6]. For this weld both as-welded (0 re-fuse) and a ‘1 re-fuse’ portions of the weld were assessed by inspecting four metallographic cross sections from each region. ‘Re-fuse’ welding is the practice of autogenously re-melting selected layers of weld metal, which results in increasing the plastic strain in the underlying weld joint. The data were further partitioned between the cold wire section of the weld (top plot) and the hot wire section of the weld (bottom plot). As shown, EN52i easily met the cold wire cracking criterion of < 20 cracks in 8 metallographic cross sections, with cracking resistance comparable to EN82H. In the hot wire portion of weld, 2 of the 3 heats met the criterion of < 100 mils of total crack length. Only heat 187775 failed to meet this target with somewhat more solidification cracking than desired. As previously noted in the transversestrain tests, heat 187775 contains higher than optimal levels of niobium and the new specification limits on Nb (2.2-2.4 vs the 2.5 wt.% measured in heat 187775) will bring the solidification cracking resistance in-line with EN82H.

Lastly, vendor experience with EN52i in a range of potential applications is summarized in Table III. Automatic GTAW, manual GTAW, and automatic GMAW processes were assessed on a range of base metals. In all cases, weldability and welding characteristics like bead flow and tie-in were judged to be as good or superior to each Vendor’s experience with EN82H. In addition to the intrinsic weldability (resistance to solidification and ductility dip cracking) that the alloy was designed for, Outokumpu has optimized the minor alloying elements and the weld wire processing in order to minimize tenacious surface oxides and promote good flow and tie-in of the weld beads [7, 8].

3.4 Environmentally Assisted Cracking

3.4.1 Low Temperature Crack Propagation

Fracture toughness data of EN52i along with comparison data for EN82H are given in Figure 8a. As shown, the toughness in air at both room temperature and 640°F is very high with $J_{IC}$ values > 1500 in-lb/in². Similar to EN82H welds, the fracture toughness is degraded by testing in high temperature hydrogenated water with toughness values dropping to < 500 in-lb/in² and the fracture mode changing from ductile failure to intergranular fracture [9, 10]. Figure 8b summarizes the EN52i data at a test temperature of 130°F as a function of hydrogen concentration. The EN52i data show generally higher toughness values than a lower bound limit for EN82H, with a significant increase in toughness as the hydrogen concentration in the water is dropped ≤ 15 scc H₂/kg H₂O.

3.4.2 Corrosion Fatigue

An example of the corrosion fatigue crack growth rate of EN52i in hydrogen deaerated water is given in Figure 9. Additionally in Figure 9, best fit lines for 304L stainless steel, Alloy 690, and EN82H are included for comparison. As shown, EN52i displays comparable crack growth rates to EN82H and A690 and superior performance relative to 304L stainless steel.

3.4.3 Primary Water Stress Corrosion Cracking

Results from stress corrosion crack growth rate tests are given in Figures 10 and 11. The results in Figure 10 are for aggressive testing conditions (680°F, 28 scc H₂/kg H₂O near the Ni/NiO transition) and are compared to best estimate data for EN82H (from a previous and extensive testing program). The benefit in crack growth rate between EN52i and EN82H is ≥ 50X. This benefit is judged to be conservative because it the EN52i samples did not fully engage along the crack front and the cracks did not extend more the one grain into the weld. Under these same test conditions, samples of EN82H are known to exhibit nearly full engagement and extend several grains into the weld. In the bottom of Figure 10, representative fractographs are given which shows the isolated pockets or SCC ‘fingers’ in the EN52i test samples. The lack of engagement and growth beyond one grain is also an issue in determining the temperature dependence of SCC, as discussed by Moss et al. in these proceedings [11]. If low temperature SCC data are adjusted to full engagement and grain to grain incubation is not accounted for,
it appears to bias low temperature rates high, resulting in an low activation energy that is inconsistent with the ~130 kJ/mol typically exhibited by Ni-Cr alloys in high purity, high temperature water [12] [13].

The ≥ 50X benefit to PWSCC resistance is further supported by the composite weld test results. As shown in Figure 11, in a 12 month test, the E-182/EN82H samples show extensive cracking with 100% engagement of the crack and > 200 mils of crack growth which readily transitioned into and grew into several weld grains. In contrast, the average crack extent of EN52i in tests up to 24 months was < 10 mils, without full engagement and without the cracks extending beyond the first weld bead diluted with EN52i. Notably, there is no significant difference in the % engagement or the extent of cracking between E-182/EN52i tests conducted for 12 months and 24 months. These observations show that SCC markedly decreases in rate in the first weld bead diluted with EN52i at approximately 24 wt.% chromium.

3.5 Metallurgical Stability

Nickel-chromium alloys can be susceptible to the development of long range ordering to form the Ni2Cr phase at temperatures <1094°F (590°C) [14-16]. Long range ordering is undesirable since it results in a lattice contraction, significant hardening, loss of ductility, and decreased toughness. As shown in Figure 12a, several alloys could be susceptible but available data indicate that formation of this phase is sluggish in complex alloys at PWR temperatures, likely requiring decades to form [16, 17]. Moreover, the phase stability is known to be influenced by common alloying additions (e.g. Mo increases while Fe decreases the solvus temperature) [16, 18, 19], complicating the understanding of what alloys may be susceptible. Accelerated tests on a model version of EN52i (Figure 12b) indicates very good phase stability with no sign of hardening to date (i.e. 3000 hour exposure between 631–878°F, (333-470°C)). Note that 3000 hours at 878°F is equivalent to ~75 years at 631°F assuming an activation energy of 147 kJ/mol [16].

4.0 CONCLUSIONS

- EN52i possesses physical properties consistent with other nickel-chromium weld filler metals. Typical of most FCC ‘austenitic’ weld filler metals, EN52i welds display significant <100> type texture. Consequently Young’s modulus displays anisotropy.

- The tensile strength of EN52i is moderate (~65 ksi) and ductility is high (%Elongation and %RA ≥ 30%). The tensile properties are insensitive to typical post weld heat treatments (~1050°F / 30 hours) and to the welding process (GTAW vs. SMAW). Similarly, the yield strength, ultimate tensile strength, and % reduction in area were very similar for ‘L’ and ‘T’ oriented samples, but the % elongation was consistently lower for the ‘T’ orientation.

- Both in-house and vendor experience with EN52i indicates very good weldability with good resistance to ductility dip and solidification-type cracking. Moreover, vendor experience confirms that welding parameters developed for EN82H can be directly used for EN52i.

- EN52i possesses a very high fracture toughness in air and is as or more resistant to Low Temperature Crack Propagation (LTCP) than EN82H. Similarly, fatigue testing of EN52i in high temperature high purity water shows crack growth rates consistent with Alloy 690 and superior to 304L stainless steel.

- The primary water SCC resistance of EN52i is far superior to EN82H. Aggressive test conditions at 680°F near the Ni/NiO phase transition indicate ≥ 50X slower rates in EN52i relative to EN82H. This benefit is likely conservative since it is based on adjusting the crack growth to 100% engagement and does not account for an incubation time for cracking to propagate into new grains. Composite specimen testing confirms this benefit as shown by SCC in the E-182 / EN52i samples markedly slowing their rate or possibly arresting in the first weld bead diluted with EN52i.

- Testing between 631-878°F (333-470°C) for times up to 3,000 hours indicated good metallurgical stability with no hardening that would suggest susceptibility to long range ordering.
5.0 ACKNOWLEDGEMENTS

The Author’s are indebted to and gratefully acknowledge the contributions of the Specialists, Technicians, and Welder’s at the Bettis and Knolls Atomic Power Laboratories who performed the testing reported in this paper, as well as our colleagues at Outokumpu VDM USA LLC who melt and fabricate Filler Metal 52i.

6.0 REFERENCES

### Table I. Comparison of the Composition of Each Heat of Material in wt.%

<table>
<thead>
<tr>
<th>Heat</th>
<th>Cr</th>
<th>Fe</th>
<th>Nb</th>
<th>Mn</th>
<th>Ti</th>
<th>S</th>
<th>P</th>
<th>C</th>
<th>N</th>
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<tbody>
<tr>
<td>MLTS-2</td>
<td>26.83</td>
<td>3.05</td>
<td>2.51</td>
<td>3.19</td>
<td>0.17</td>
<td>0.0014</td>
<td>0.003</td>
<td>0.032</td>
<td>0.009</td>
</tr>
<tr>
<td>187775</td>
<td>26.98</td>
<td>2.55</td>
<td>2.58</td>
<td>3.04</td>
<td>0.37</td>
<td>0.0010</td>
<td>0.002</td>
<td>0.040</td>
<td>0.039</td>
</tr>
<tr>
<td>126373</td>
<td>26.97</td>
<td>2.55</td>
<td>2.29</td>
<td>2.97</td>
<td>0.30</td>
<td>0.0010</td>
<td>0.003</td>
<td>0.043</td>
<td>0.017</td>
</tr>
<tr>
<td>Specification*</td>
<td>26.0</td>
<td>2.0</td>
<td>2.2</td>
<td>2.5</td>
<td>0.2</td>
<td>&lt;0.002</td>
<td>&lt;0.008</td>
<td>0.03</td>
<td>0.015</td>
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*Most recent values based on current testing

### Table II. Summary of Selected Room Temperature Physical and Thermal Properties of Filler Metal 52i as well as the Approximate Melting Range

<table>
<thead>
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<th>Property</th>
<th>MLTS-2</th>
<th>187775</th>
<th>126373</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, $\rho$</td>
<td>0.296 lb/in³</td>
<td>8.19 g/cm³</td>
<td></td>
</tr>
<tr>
<td>Mean Coefficient of Thermal Expansion, $\alpha$</td>
<td>6.8x10^{-6} (in/in/°F)</td>
<td>1.2x10^{-5} (cm/cm/°C)</td>
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<tr>
<td>Specific Heat, $c$</td>
<td>0.106 (Btu/lb-°F)</td>
<td>444 (J/kg-°C)</td>
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<tr>
<td>Thermal Conductivity, $\lambda$</td>
<td>6.0 (Btu/ft-hr-°F)</td>
<td>10.4 (W/m-K)</td>
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<tr>
<td>Thermal Diffusivity, $\kappa$</td>
<td>4.4x10^{-3} (in²/sec)</td>
<td>2.8x10^{-6} (m²/sec)</td>
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<tr>
<td>Melting Range</td>
<td>2380°F – 2490°F</td>
<td>1304°C – 1366°C</td>
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</table>

### Table III. Summary of Industrial Experience with EN52i

<table>
<thead>
<tr>
<th>User</th>
<th>Process</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>B&amp;W (Barberton)</td>
<td>Automatic GMAW and manual GTAW cladding and groove welds</td>
<td>EN52i can directly replace EN82H</td>
</tr>
<tr>
<td>B&amp;W (Mount Vernon)</td>
<td>Automatic GTAW cladding</td>
<td>EN52i has similar weldability, bead texture, bead tie-in, and puddle control as EN82H</td>
</tr>
<tr>
<td>Hamill Manufacturing</td>
<td>Manual GTAW for dissimilar metal weld joints</td>
<td>EN52i has the same welding characteristics as EN82H</td>
</tr>
<tr>
<td>Electric Boat</td>
<td>GTAW pipe welds with Alloys 600 and 690</td>
<td>Excellent flow and tie-in with EN52i</td>
</tr>
<tr>
<td>Newport News Shipyard</td>
<td>GTAW pipe welds with Alloys 600 and 690</td>
<td>Excellent flow and tie-in with EN52i</td>
</tr>
<tr>
<td>Arc Applications</td>
<td>GTAW steam generator tube plug development</td>
<td>No significant difference between EN52i and EN82H</td>
</tr>
</tbody>
</table>
Figure 1. The temperature dependence of the density, mean coefficient of thermal expansion, specific heat, thermal conductivity, and thermal diffusivity of filler metal EN52i.
Figure 2. Comparison of Poisson’s ratio from room temperature tests in the L, S, T and diagonal ‘D’ orientations. The average value of all the tests is $\nu = 0.26$.

Figure 3. a.) Illustration of crystallographic texture in GTA welds and b.) typical elastic modulus values in the ‘L’, ‘S’, and ‘T’ orientations, as well as off axis diagonal samples. As expected, <100> texture in the L, S, and T directions results in lower Young’s modulus.
**Figure 4.** Comparisons of the typical tensile properties for EN52i a.) as-welded vs. a 1050°F / 30 hour stress relief, b.) gas-tungsten-arc vs. shielded-metal arc welds and c.) the ‘L’ (welding direction) vs. ‘T’ (transverse) orientations. Note the relatively consistent properties with little effect of stress relief, welding process, or orientation. The one exception to this is the ‘T’ direction % elongations that were consistently lower than the ‘L’ tests (4c)
**Figure 5.** a.) Summary of transvarestraint weldability data and b.) examples of solidification cracking at 5% strain for select alloys. Note that the data for EN52i, especially heats 126373 and MLTS-2 lay relatively close to EN82H.
A-GTAW
Dissimilar Metal Weld

SMAW
Dissimilar Metal Weld

Figure 6. Summary of weldability testing on a dissimilar metal (carbon steel to stainless steel) v-groove weld for automatic GTAW (top) and SMAW (bottom). Note the good performance of EN52i relative to other high chromium weld filler metals.
A-GTAW
Cold Wire Portion of
Narrow Groove

A-GTAW
Hot Wire Portion of
Narrow Groove

Figure 7. Summary of weldability testing on an automatic gas-tungsten-arc narrow groove weld. The data were assessed in the cold wire region (top plot) and the hot wire region (bottom plot). Note the good performance of EN52i relative to other high chromium weld filler metals.
Figure 8. a.) Comparison of the fracture toughness of EN52i with EN82H in air and hydrogenated water and b.) the effect of hydrogen concentration in the water on the elastic-plastic fracture toughness of EN52i. Note that EN52i displays comparable or better toughness than the baseline heat of EN82H.

Figure 9. Illustration of the corrosion fatigue performance of EN52i relative to stainless steel (long dash black line), Alloy 690 (short dashed red line), and EN82H, (dashed dot purple line).
Figure 10. Comparison of the best estimate stress corrosion crack growth rates of EN82H and EN52i at 680°F (360°C) as a function of stress intensity factor (top graph). The ≥50X benefit of EN52i is judged to be conservative since the EN52i rates are adjusted to full engagement and do not take into account any incubation time to propagate a crack into different crystallographic grains. The light microscopy shows typical isolated pockets or ‘fingers’ of SCC.
Figure 11. Summary of composite material SCC testing. While SCC readily grows and extends from E-182 into EN82H (left plot and picture), it grows much slower or possibly arrests in the first weld bead diluted with EN52i (right hand figure and pictures). Note that there is no apparent difference in crack engagement or extension between 12 month and 24 month exposures of E-182/EN52i.

Figure 12. a.) Illustration of where some commercial nickel-chromium alloys lie in relation to the Ni<sub>2</sub>Cr phase estimated by Xiong [17] and b.) isothermal exposures of EN52i compared to a Ni-30Cr binary alloy. The dashed lines show how the binary alloy hardens due to long range ordering while the lack of hardening in EN52i (data points) indicate good metallurgical stability.