FRACTURE TOUGHNESS INVESTIGATIONS OF LONG-TERM BEHAVIOUR OF
G-X 8 CrNi 12 CAST STEEL

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In order to study the long-term toughness behaviour of G-X 8 CrNi 12, samples obtained from the large test block were subjected to the long-term aging at temperatures of 300°C and 400°C; aging times up to 30,000 hours. The mechanical, brittle fracture and fracture mechanical properties do not change during the aging at the temperature of 300°C. Specimens exposed to the temperature of 400°C did show certain embrittlement characterized by the decrease of the impact value, fracture toughness and transition temperatures. Results obtained were confirmed by the metallographic and SEM examinations.

INTRODUCTION

G-X 8 CrNi 12 type cast steel is characterized by its resistance against erosion and corrosion as well as by its satisfactory welding properties. The material is standardized according to DIN 17245 and the VdTUV-material form 442.11/81; it can be used for pressure vessels and steam boilers up to operating temperatures of 320°C. When utilizing this material for e.g. inner casings in saturated steam turbines up to operating temperatures of less than 300°C (1), it is to be guaranteed that the mechanical and fracture mechanical properties having been achieved as delivered won’t be essentially impaired even in service.

Problems concerned the martensitic chromium cast steel e.g. G-X 5 CrNi 13 4 were referred in the literature many times already (2,3,4). There was the long-term aging behaviour in temperature range from 250°C up to 500°C studied and the decrease of the toughness of the steel, exposed to the temperatures of above 350°C found. The considerable embrittlement determined at the temperatures of above 400°C up to approximately of 500°C can be explained as the "475°C embrittlement", caused by successive precipitation of chromium in ferrite. It is at present not yet clear whether the same embrittling mechanism is also responsible for the decrease of the toughness of the steel, being long-term exposed below 400°C (2), or whether it happens an additional changing of segregations along the primary grain boundaries or precipitations in the grains.

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Furthermore these steels can embrittle during the cooling from tempering temperature because of the so called "temper embrittlement". The reason for that are residual elements in interaction with alloying elements which segregate on grain boundaries.

The purpose of this investigation was to learn about the behaviour of the material G-X 8 CrNi 12, having \( \frac{3}{4} \) % nickel only, being long-term exposed at temperatures of 300 and 400°C, and to determine whether and after which exposure times the decrease of the toughness has to be taken into account in order to enable the assessment of the service behaviour of long-term thermally stressed components.

**TEST MATERIAL AND METHOD**

The study was carried out on a round test block of about 450 mm diameter and 450 mm in height, cast from the steel 0-2 8 CrNi 12, molten in electric arc furnace. The chemical composition and the actual heat treatment are given in Table 1 and 2.

The usual heat treatment of casings of this steel grade includes a thermal step, causing a partial austenitic transformation, which leads to an increase of toughness. In this case the modifications of the usual heat treatment procedure, i.e. the omitting the partial austenitisation and prolonged holding times, were decided in order to compensate the heat treatment steps, connected with the welding on casings and to obtain the so called "over-aged"-state of the material (worst case).

In order to investigate long-term toughness drop, samples for CTE-specimens were taken from the block center and exposed to temperatures of 300°C resp. 400°C up to 30,000 h, corresponding to about 4 years of service. Up to times of 3,000 h only the hardness and notch impact energy of ISO-V-specimens were measured at room temperature. After aging periods of 5,000, 10,000, 20,000 and 30,000 h the mechanical properties, the impact energy transition temperature curve with the fracture transition temperature FATT, the NDT-temperature as well as the fracture toughness at -60 or -50°C were determined. These low test temperatures had to be selected to determine \( K_c \)-values according to ASTM-E 399 requirements. In the following the properties having been measured on material in the as received condition are to be compared to those obtained on the material after long-term aging exposure.

**TEST RESULTS**

**Mechanical Properties**

The mechanical properties of the material in the as received conditions were determined on tensile test specimens, taken from the position near the surface and from the center of the test block. There was no influence of the location of the specimen on the properties observed.

Fig. 1 illustrates the influence of aging on mechanical properties at room temperature. Both, the 0.2-yield and tensile strength are not influenced by aging at 300°C and 400°C. The reduction of area \( A \) and elongation \( A \) increase with prolonged exposure times. The exact explanation for this tendency could not be given in this study. The hardness value HB 5/750 was found in the range of 180-200, the value was not influenced by aging.

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Impact Energy Temperature Behaviour and Fracture Transition Temperatures

Fig. 2 shows impact energy temperature curves of material in an as-received condition. The transition region extends from about -40 to +60°C with fracture transition temperature FATT of about +20°C. The upper shelf at 0% crystallinity will be reached at about +80°C. Similar as in case of the mechanical properties mentioned above the location of impact specimens did not show an influence on impact values, when the scattering of the results is taken in account. In order to determine the relevant loss of ductility in the as-received condition due to possible temper embrittlement in addition "dembrittlement" and "embrittlement" heat treatment was performed. The "dembrittlement" heat treatment (heating up to just below the temper temperature and quenching) caused an increase in toughness. After the "embrittlement" heat treatment, however, (after heating and cooling off in the furnace in a period of 160 h) only a slight variation compared to the as-delivered state was established; i.e., that in the as-delivered state - simulated by heat treatment - an additional temper embrittlement took place.

Fig. 3 shows the results after aging. There was no variation of the impact energy temperature behaviour measured after aging at 300°C for up to 30,000 h. The individual results fit in the scatter range of the as-received state. Aging at temperatures of 400°C, exposure time of 5000 h and more, is characterized by a significant shift of the impact energy temperature curves in the direction towards the higher temperatures.

These variations will be even more distinct when regarding the impact energy at ambient temperature and the fracture transition temperatures. Considering the scattering of the impact energies of the as-received state, attained at the measurements on 16 impact specimens, no variation of the impact energy at room temperature could be established after an aging period of up to 30,000 h at 300°C, as shown in Fig. 4. The same applies to the fracture transition temperatures FATT and NBDT illustrated in Fig. 2.

On the other hand, in case of an aging temperature of 400°C already after 1000 h a slight decrease in toughness is to be recognized, even more evident with the FATT. Increasing aging periods are accompanied by a continuous decrease in toughness. No influence of the aging process on the upper shelf notch impact energy (0% crystallinity) - Fig. 5 is giving an example at 300°C test temperature - could be established yet. Because of the scattering of the results there is no prediction of tendency possible.

Fracture Mechanical Properties

Fig. 6 illustrates the influence of aging on fracture toughness KIc. Fracture toughness KIc after aging at a temperature of 300°C results in somewhat higher values at a test temperature of -50°C, varying in no way at a test temperature of -80°C. Aging at 400°C causes a definite decrease in fracture toughness beyond about 5000 h.
Metallographic and Micro-Fractographic Investigations

The microstructure shown in Fig. 7 is characterized by tempered martensite with primary grain sizes of 1 to 3 per ASTM. There is no substantial difference between the structure of the as received state and after aging at 300°C, observed on the usual metallographic microscope. But after an aging at 400°C and 30,000 h signs of diffusion in form of a decomposition of the martensitic structure along the primary grain boundaries are to be seen.

Microfractographic examinations of fractured surfaces of impact specimens in as received condition (broken at ambient temperature, characterized by the impact energy of 80 J and crystallinity of 50 %) revealed in the area of the crystalline fracture a mixed fracture consisting of dimple and cleavage fracture with intergranular fractures, Fig. 8.

The fracture surfaces of CT-specimens tested at -80°C show a similar appearance in the area of unstable crack extension (Fig. 9). Surprisingly, also nearby the fatigue crack, intergranular crack propagation could be observed. The intergranular crack propagation, observed in samples in the as received condition, indicates that in this state a certain embrittlement took already place, caused by the so called "over-aged" treatment of this material and by additional temper embrittlement.

Specimens examined in the "deembrittled" conditions did show in the center of the crystalline fracture area predominantly transgranular quasi cleavage fracture and only very slight intergranular fracture portions.

The fracture surface of the impact specimen exposed 30,000 h at 300°C (Fig. 10) and tested at room temperature differs in no way from the state as received (Fig. 8). The same fracture surface features are to be observed in both cases. Already after an exposure of 5,000 h at 400°C a significant increase in intergranular fracture proportions in the area of crystalline fracture could be observed. It is extending across 90 % of the total fracture surface of the impact specimen and after the exposure of 30,000 h being exclusively characterized by an intergranular fracture as shown in Fig. 11 above. Thus, the loss of toughness is clearly reflected by the intergranular crack propagation appearance.

A modification of the precipitation structure on the grain boundaries caused by aging could not be observed. If the toughness decrease after aging is induced by similar mechanisms as during the "475°C embrittlement" a change of the microstructure in the interior of the grain will take place. In chromium steels with higher nickel content the change causes an increase in hardness and strength and a decrease in the upper shelf energy. In the described investigation these phenomena could not be measured. Thus, it can be concluded that in this type of steel the microstructural modification takes place with a slower velocity.

Impact specimens of the upper shelf region will show a pure dimple fracture (Fig. 11 at the bottom) even after 30,000 h aging at 400°C.

In case of ductile fractures crack initiation is predominantly influenced by precipitations within the grains.
When considering that aging does not cause essential changes in this microstructure, aging can not become effective in the formation of the ductile fracture. Therefore, aging need not cause any variation of the upper shelf toughness.

DISCUSSION OF RESULTS AND CONCLUSIONS

The brittle fracture, fracture mechanical and microfractographic investigations have clearly shown that aging at 300°C and up to 30,000 h will not influence the determinative toughness characteristics. Aging at 400°C, however, results in measurable losses of toughness already starting with about 1,000 h. The transition temperature curve will be shifted towards higher temperatures with increasing aging; fracture transition temperatures FATT and NDTT are rising. The impact energy at room temperature, characterizing the transition region of the impact energy temperature curve as well as the fracture toughness of the lower shelf decreases.

The components manufactured out of the relevant cast steel will not normally be used at 400°C; accordingly, these particular toughness losses need not be taken into consideration. On the other hand, the aging at a temperature of about 300°C for the period of 30,000 h does not show any distinct influence of the long-term exposure on the toughness values. A prediction of the behaviour of the aged material can be given on the basis of a diagram, presented by Trautwein and Gyzel (3,4), which was prepared for steels, having 20 % Cr and 8 % Ni or 15 % Cr and 4 % Ni. It is based on investigation results, illustrating that the aging behaviour of chromium alloyed steels is generally determined by the chromium diffusion within the lattice. As measurements have shown, its activation energy is essentially independent on the temperature in the 300 to 400°C range at hand. The resulting physical equivalence between aging time and temperature enables a complete illustration of all the results and predictions regarding the aging behaviour at time and temperature conditions outside of the measuring range.

The combined influence of time and temperature may be described by means of aging parameter P, with reference point P = 1 corresponding to an aging of 10 h at 400°C; it may be calculated according to the following equation for arbitrary aging conditions:

\[ P(t, T) = \frac{1}{t} + 0.4343 \frac{U}{R} \cdot \left( \frac{1}{673.2} - \frac{1}{T} \right) \]

\( t \) - time of reaction in hours (exposure time)

\( T \) - temperature of reaction in deg K (aging temperature)

\( R \) - gas constant = 2 cal mol deg K

\( U \) - activation energy = 24,000 cal/mol

This relation was experimentally confirmed for steels 15 % Cr 4 % Ni and 20 % Cr 8 % Ni. The aging results and the aging parameter P determined for the relevant aging conditions are summarized in the aging diagram.
Fig. 12 is showing impact energies, determined at room temperature for G-X 8 CrNi 12, in such a diagram. The parameter $P$, relating to the relevant aging temperature and aging time can be obtained as shown in the detail, see Fig. 12 below.

As an example: The aging at 400°C for 1,000 hours is characterized by the parameter $P = 3$. This parameter corresponds to the aging of approximately 22,000 hours at 300°C. This value will be nearly equivalent to the threshold value, marking the range, from which the toughness losses due to the aging at 400°C have to be taken in account.

Should the above mentioned relation be applied for G-X 8 CrNi 12, would the aging at 300°C for 100,000 hours, described by $P = 3.65$, cause the decrease of the impact energy from the 86 J in the delivered condition on the value of about 45 J. This assessment has to be confirmed by other experiments. Furtheron, the fracture toughness can be determined empirically by means of Harnon-Rolfe equation (5,6) on the base of the relevant impact energy. The value obtained for the "as received" condition at room temperature is $K_{icav} = 4,600$ N/mm².

This value corresponds to values of fracture toughness determined on various melts from this steel grade (1). The fracture toughness value of this cast steel, calculated for conditions of aging at 300°C for 100,000 hours, would be $K_{icav} = 3,300$ N/mm².

Such a value exhibits a loss of fracture toughness of about 30 %, nevertheless it represents a value which is usually determined for ductile ferritic cast steels (1,7).

Summarizing it may be stated that with respect to the results at hand the long-term behaviour of G-X 8 CrNi 12 is similar to that one of the higher alloyed chromium-nickel steels (like G-X 5 CrNi 13 4) and obviously can be described by means of the aging parameter $P$. It is evident that certain decrease of the toughness caused by the long-term exposure to temperatures up to 300°C has to be taken in account, which is compensated by the relatively high initial toughness in the "as received" condition.

REFERENCES

Figure 1 Effect of aging on mechanical properties

Figure 4 Effect of aging on impact energy

Figure 2 Impact energy-temperature-curve as received

Figure 5 Effect of aging on transition temperatures FATT and NDTT

Figure 3 Impact energy-temperature-curve after aging

Figure 6 Effect of aging on fracture toughness
Figure 7: Influence of aging temperature on microstructure

Figure 8: Fracture surface of impact specimen - as received

Figure 9: Fracture surface of compact tension specimen - as received

Figure 10: Fracture surface of impact specimen after aging 300°C/30,000 h

Figure 11: Fracture surface of impact specimen after aging 400°C/30,000 h

Figure 12: Effect of aging on impact energy
### TABLE 1 - Chemical Composition in %

<table>
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<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>N</th>
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<td>DIN 17 245</td>
<td>0.06-0.10</td>
<td>0.10-0.40</td>
<td>0.50-0.80</td>
<td>&lt; 0.035</td>
<td>&lt; 0.025</td>
<td>11.5-12.5</td>
<td>&lt; 0.50</td>
<td>&lt; 0.50</td>
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<td>Test Block</td>
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<td>0.20</td>
<td>0.49</td>
<td>0.007</td>
<td>0.010</td>
<td>12.3</td>
<td>0.04</td>
<td>1.17</td>
</tr>
</tbody>
</table>

### TABLE 2 - Test Treatment

**Recommended:**
1050°C/air < 100°C + 780...850°C/h/air < 100°C + 650...720°C/h/air or air.

**Test Block:**
1050°C/24 h/ventilator + 750°C/45 h/furnace up to 200°C.

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