# DETERMINATION OF THE MICROSTRUCTURAL CREEP PROPERTIES OF CAST IRON CONNECTOR AT HIGH TEMPERATURES FOR THE PREDICTION OF THE THERMO-MECHANICAL BEHAVIOR OF ANODIC ASSEMBLIES

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#### Abstract

The prediction of the air gap at the cast iron to carbon interface in the anodic assemblies as well as its closing and pressure build-up during the operation remain a complex task. It is therefore very difficult to predict the electrical performance of anodic assemblies. This main difficulty comes from the evolution of the thermophysical and thermo-mechanical properties of the microstructure mapping during these stages. To overcome this difficulty, a natural approach is to characterize this mapping of microstructures.

In this paper, the creep properties at high temperature of the cast iron connector are determined using this approach. The results show highest creep strength of the pearlitic and white cast irons below  $475-550^{\circ}$ C compared with the ferritic microstructure. With the temperature, the creep deformation increases but above  $550^{\circ}$ C a graphitization in the pearlitic and white cast irons contributes to their growth, which is comparable or exceeds the creep rate.

## Introduction

This investigation was undertaken to characterize the time dependent deformation of the industrial cast iron connector. At high temperatures, a creep of cast irons decreases stronger than the yield strength [1, 6] and begins to play a certain role in the thermomechanical behavior of the anode assembly, and particularly, in the evolution of the air gap between carbon block and cast iron connector.

The chemical and microscopic analyses of the industrial cast iron connector indicate that depending on the chemical composition, diffusion of elements and different cooling conditions, the carbon equivalent has the average range 4.26-4.38, the grey and/or white cast iron can be formed. At the boundary with the steel stub and/or carbon block there is the white and/or mottled cast iron depending on wall thickness and shape of the connector. The central zone of the cast iron connector consists mainly of the fully pearlitic microstructure. The zone of the ferritic microstructure with the undercooled graphite is located between the white and/or mottled cast iron and central pearlitic zone. Such a map distribution is observed over the entire height of the connector but with various ratios between microstructures depending on wall thickness and its shape.

The cast iron samples with defined microstructures were produced as a demonstration part of the industrial cast iron connector and used for creep tests to evaluate the influence of the microstructures on the creep deformation of the cast iron connector.

The creep was defined as time dependent plastic deformation at constant stress and temperature. The form of a typical creep curve of strain versus time is shown in Figure 1 [2]. The slope of these curves is the creep rate (secondary creep) that was used to estimate

the probable deformation throughout the life of the cast iron sample. Testing either at higher stresses or higher temperatures will increase the steady-state creep rate and reduces the rupture lifetime as illustrated in Figure 1 [2]. The curves may show the instantaneous elastic and plastic strain that occurs as the load is applied, followed by the plastic strain which occurs over time.

The minimum secondary creep rate is of the most interest to design engineers, since failure avoidance is required and in this region some predictability is possible. In secondary (Steady State) creep [2] there is a balance between work hardening and recovery processes, leading to a minimum constant creep rate. Secondary creep, which occurs as a linear function of time, is strongly dependent on temperature and stress. Since creep is a thermally activated process, the minimum secondary creep rate can be described by a fundamental Arrhenius equation of the form:

$$\dot{\varepsilon}_{ss} = \dot{\varepsilon}_{mn} = \mathbf{A}\sigma^n \exp\!\!\left(\frac{-\mathbf{Q}}{RT}\right)$$

where *n* is the stress exponent, Q is the activation energy for creep, R is the universal gas constant and T is the absolute temperature. Log-log plots of minimum creep strain rate versus applied stress (see Figure 2) often result in a bilinear relation in which the slope, n, at low stresses is equal to one indicating pure diffusion creep, and n at higher stresses is greater than one, indicating power law creep with mechanisms other than pure diffusion (e.g., grain boundary sliding) [2].

The goal in engineering design for creep is to predict the behavior over the long term. There are three key methods: stress-rupture, minimum strain rate vs. time, and temperature compensated time. No matter which method is used, two important rules have to be observed:

- 1. The secondary creep test time have to be at least 10-15% of design time. Average operational time of the connector is about 25 days (600 hours). In this connection, the time of each creep test was 144-384 hours (6-16 days) to obtain the straight line of the secondary (steady state) creep.
- 2. The creep mechanism must not change with time, temperature or stress.

# Materials and experimental procedures

The presented microstructures were determined in the industrial cast iron connector: the pearlitic microstructure in the central part of the connector (see Figure 3a) takes 30-50% depending on the wall thickness; the ferritic (80-90%) - pearlitic (10-20%) microstructure with the undercooled graphite in the ferritic zone (see Figure 3b) takes 40-45%; mix of the white/mottled (50-100%) cast irons and pearlitic (0-50%) cast iron (see Figure 3c) takes about 5-25% of the wall thickness and located near carbon block and steel stub.



Time Figure 1. The typical creep curves [2].



 $\log \sigma$ 



a) Pearlitic microstructure

b) Ferritic-Pearlitic microstructure

c) Mix of the white/mottled cast irons and pearlitic microstructure.

Figure 3. The as-cast microstructures of the cast iron connector.

The cast iron samples (D25xH35mm)of the industrial chemical with composition the corresponded microstructures produced were by "Laboratoire de Mécanique de Lille" (France) and the plant of RioTintoAlcan in Alma (Canada), and machined by University Research Centre on Aluminum (CURAL).

The creep tests were conducted in air under conditions of constant stress with the temperature continuously monitored and controlled within  $5^{\circ}$ C of the desired value. The strain of the specimens was measured with two linear variable differential



Figure 4. The installed LVDTs.

transformers (LVDTs), which were installed outside the furnace (see Figure 4). The top and bottom heads of the creep-testing machine were cooled by cold water to limit the heating and decrease the measurement errors.

The creep tests were performed at the temperatures 400-800°C and equivalent stresses 30-60MPa for these temperatures reflected the service conditions and calculated using a numerical simulation software. For each temperature, the range of the stresses was corrected according to the condition of obtaining of the secondary (steady state) creep curve.

The samples were preloaded/strained at the load 0.5kN and room temperature prior to creep testing and heating to decrease the influence of the contact between loading heads and samples, and to simulate the real conditions. During heating (10°C/min) the value of preloading was being kept at the same initial level to take into account the influence of the thermal expansion of the sample, top and bottom heads. After reaching the necessary temperature, the sample and heads were being soaked for 2 hours to decrease the influence of theirs thermal expansions. After soaking, the stress (2MPa/sec) on the specimen was increased incrementally and held 144 hours (ferritic and pearlitic microstructures) and 388 hours (mix of white and pearlitic cast iron). The results are represented on the screen (see Figure 5).



The creep tests were conducted under conditions of Standard E139-

00, ASTM "Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials" [3].

#### The results and discussions

During the first preliminary creep tests, it was established that stresses > 50MPa at 700°C or stresses > 75MPa at 600°C lead to the fast plastic deformation of the industrial cast iron with any microstructure and its failure. The strain 32% (maximum value measured by the installed LVDTs) of the cast iron with ferritic microstructure (Fig.3b) is reached at 50MPa and 700°C after 60 hours; 1.2 hours need at 75MPa and the same temperature; stress 125MPa produces this strain for 3 minutes. Such behavior of the cast iron in this high stress and temperature region is described by the high power law creep and dominated by the primary creep when the creep rates are exceptionally fast and the values of *n* are large.

In steady-state creep when the strain increases linearly with time, the creep behavior shows the typical appearance of metal matrix with a tendency for the apparent stress exponent n < 5 or  $n \approx 5$  (see Figure 6). Calculation of the stress exponent n for other microstructures showed that theirs creep nature is complied with the power law creep, n = 3-6. Under these conditions, the minimal creep rate can be defined accurately using the plots of the total strain *vs* time on Cartesian coordinates.

The creep curves for the different microstructures were built and used to calculate the minimum creep rate for each case at the specific temperature and stress. It was done by drawing a tangent to the curve at the minimum slope of the steady-state creep.

The influence of the stress levels on the minimum creep rate is shown in Figure 7. Three graphs for each microstructure were built to evaluate their creep behavior and determine the minimum creep rate, which increases with the stress levels and temperature, giving a linear relationship. As the temperature increases, the stress level that gives the minimum creep rate reduces accordingly. The extrapolation of the curves for each microstructure in the plots of stress *vs* creep rate to a minimum creep rate permits us to determine the stress, which produces this rate at the certain temperature. For instance, it was established for ferritic microstructure (see Figure 7a) that the increase of the temperature by each  $100^{\circ}$ C decreases the stress (which produces the creep rate of 0.0001%/h) in about 2 times. The scale was removed for reasons of confidentiality.



500°C (35-75 MPa): n=4.0	600°C (10-35 MPa):	n=4.6
700°C (10-35 MPa): n=4.7	800°C (5-15 MPa):	n=5.6





The nature of the curves on Figure 7, the range of data and some values are accorded with the literature results of the creep test of similar cast irons. The obtained results can be used to determine the creep rate for each microstructure of the industrial cast iron connector depending on the applied stress and temperature that permits to supplement the numerical model of the thermomechanical behavior of the anodic assembly. These graphs were replotted in the stress-temperature coordinates and presented on Figure 8 for the creep rates: 0.001%/h and 0.01%/h.

The comparison of the graphs on Figure 8a and Figure 8b allows us to define the critical temperatures at which the curves for the different microstructures are intersected. Below 500-550°C the experimental and literature results indicate that the pearlitic and white cast irons have a higher level of stress-creep properties than the ferritic cast iron (see Figure 8a,b). However, above 550-600°C, the drop of the stress-creep properties of the pearlitic and white cast irons is essentially more than the drop of the ferritic microstructure creep properties; and with the temperature, the total deformation of the pearlitic and particularly white cast irons becomes bigger or similar to the deformation of the ferritic matrix. Regarding to the behavior of the cast iron connector, it means that at the elevated temperatures the graphitization in the pearlitic and white cast irons contributes to the growth of these cast iron types, which is comparable or exceeds the creep rate. The thermal expansion of the cast iron connector's part, which contain the pearlite, has the straight-line character below 600°C, but above this temperature theirs thermal expansion increases faster (than the thermal expansion of the ferrite matrix) due to a decomposition of the eutectoid cementite and correspondingly the rapid increasing of the specific volume (see Figure 8a, curve W) [8]. Than less content of pearlite in the microstructures the lower thermal expansion of the cast iron.

The further heating of ferritic/pearlitic/white cast irons between 800-860°C and 900°C leads to the increasing of the thermal expansion due to the significant growth of the austenite grain and the homogenization of the austenite due to graphite dissolution [4,6,7].

It should be noted that the temperature of the beginning cementite decomposition significantly depends on content of Si. At 3.6%Si the decomposition starts at 620°C, the lowering to 2.5%Si leads to an increase the temperature up to 630°C. With 1.7%Si the process is stalled and the decomposition starts only at 705°C, the degree of graphitization in this case is reduced to 15-20% [4,6,7].

The comparison of the graphs on Figure 8a and Figure 8b allows us also to evaluate the influence of the stress on the creep rate at the same temperature. For instance, in the ferritic microstructure at the temperature of 650°C the increase of the stress in about 1.5-2 times causes the increase of the creep rate in 10 times.

In the white cast iron, the increase of the temperature from  $650^{\circ}$ C up to  $700^{\circ}$ C (see Figure 8a) at the certain minimal stress level leads to the additional growth of the cast iron due to the thermal expansion, which is bigger than the deformation caused by the minimal creep rate of 0.001%/h. The significant increase of the stress at the same temperature 700°C contributes to the increase of the creep rate in 10 times up to 0.01 %/h that exceeds the growth of the cast iron due to the thermal expansion (see Figure 8b).

The additional investigation of the curves at the different creep rates 0.0001-0.1%/h helped to establish that at the providing of the certain minimum stress level, when the creep rate is bigger than the thermal expansion growth, the creep deformations of the cast irons

with different microstructures at the high temperatures >700-750°C (see Figure 8b) become similar.

A summary of own experimental results and literature reviews [8-10] for the cast irons show that above  $550-600^{\circ}$ C, unalloyed grey ferritic–pearlitic and white/mottled cast irons cannot provide good dimensional stability for many engineering designs, where high creep strength is required, due to theirs scaling and significant growth (especially, of the white cast iron). In the industrial cast iron connector, the arising stresses at the elevated temperatures can create such conditions when stress exponent *n* reaches the value of 5 that corresponds to the power law creep nature and high creep rates.

Literature review [2, 5] also demonstrates that the use of the ferritic or pearlitic ductile cast irons sometimes permits to change the creep nature or increase the stress level on 10-30%. The comparison of the experimental results and literature indicates the beneficial effects of Ni, Cr or/and Mo additions on the stress-creep (rupture) properties of grey cast irons at all temperatures. Adding Ni to an unalloyed cast iron or up to 1-2% Mo continuously raise the stress up to 2-4 times at all temperatures.



Figure 8. The correlation between stresses and temperatures for the ferritic (F), pearlitic (P) and white (W) cast irons at the creep rates: a) 0.001%/h and b) 0.01%/h.

### Conclusions

1. The creep properties of the cast iron change considerably during the heating and operational stage of anode assembly. Noticeable creep deformation with power law creep nature occurs at the low stresses and temperatures below 500-600°C, but at the high temperatures the creep rate begins to accelerate with time, and the material enters in the tertiary stage that leads to creep rupture.

2. At the elevated temperatures  $>600^{\circ}$ C, the increasing of the initial stress shortens the period of time spent in each stage of creep and significantly increases the creep deformation. At the same time, at the elevated temperatures, the influence of the different microstructures becomes weaker, the creep strengths of the pearlitic and white/mottled cast irons become similar or lower the creep strength of the ferritic cast irons.

3. It is very important to distinguish a phenomenon of growth and creep deformation at the elevated temperatures for the pearlitic and white/mottled cast irons. At the heating  $>700^{\circ}$ C (see Figure 8), the deformations, which are connected with the thermal expansion of these materials, can be comparable or exceed the deformations admissible at the evaluation of the creep due to the rapid increasing of the specific volume that is connected with the decomposition of the eutectoid cementite and growth of the austenite grain.

4. The determination of the creep properties of the industrial cast iron connector with different microstructures is the part of the project about the investigation of the thermo-mechanical behavior of the anode assembly. The determined relations between the stresses, temperatures and creep rates for the different microstructures of the industrial cast iron connector have to be included into the numerical model of the thermo-mechanical behavior of the anode assembly to predict the air gap at the cast iron to carbon interface.

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