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### Materials Science & Engineering A



journal homepage: www.elsevier.com/locate/msea

## Creep behavior accompanying oxidation of compacted graphite cast iron

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ARTICLE INFO	A B S T R A C T				
A R T I C L E I N F O Keywords: Compacted graphite cast iron Creep deformation Creep fracture Creep-oxidation crack Cementite decomposition	The creep behavior accompanying oxidation of a compacted graphite cast iron (CGI) containing Cu, Mo and Sn elements was studied at temperatures ranging from 350 °C to 550 °C and creep loads ranging from 40 MPa to 150 MPa. The creep damage was observed using both optical and scanning electron microscopy (SEM). The results showed that obvious creep deformation occurred when $T/T_m > 0.5$ ( <i>T</i> is testing temperature, $T_m$ is melting temperature of the CGI) and the load is higher than 150 MPa. The creep deformation in specimen was accounted from three parts: (I) deformation of matrix, (II) initiation and propagation of creep cavities and (III) debonding between graphite and matrix. In addition, two models of crack were observed during creep deformation: (I) cracks propagated preferentially in ferrite phase and combined with debonding between graphite and matrix and (II) creep microcracks caused by creep cavities in the grain boundary. These two types of crack are usually accompanied with oxidation and the former one is more harmful than the latter one because of high crack propagation rate accelerated by interfacial debonding indirectly. Finally, the mechanism of creep deformation and fracture was discussed				

#### 1. Introduction

The design of high temperature components experiencing cyclic viscoplasticity and time dependence is nowadays a major scientific issue in an industrial point of view. Reliability has become a crucial point in the automotive industry as it is now necessary to offer the most reliable components and machines to customers in a very competitive market. With increasing of combustion pressure and rotation speed of crankshaft, in other words, the service conditions of combustor chamber components, in particular, cylinder head become severe [1–3] with increment of power density of diesel engine. Compacted graphite cast iron (CGI) alloys have been attracting interesting of researches because of its excellent combination of mechanical and physical properties [4,5]. Therefore, CGI is becoming the main manufacturing material of cylinder head in advanced diesel engine [6–9] and there is increasing number of research papers on the microstructure, mechanical and physical properties and physical properties of CGI in the last two decades.

According to work by Selin [10], the strength and Young modulus of CGI is irrelevant to temperature below 300 °C. However, the strength and Young modulus decrease with the increment of temperature when temperature exceeds 300 °C. Meanwhile, the thermal conductivity of CGI is achieving the highest value at 300 °C. Y. Qiu et al. [11,12]have analyzed the microstructure evolution and deformation mechanism of

CGI at high temperature. The dislocation movement will be inhibited during material deformation and the vacancy diffusion is becoming obvious with increment of temperature. In addition, cementite can decompose to carbon and ferrite, leading to the decrement of strength and increment of plasticity. Ma et al. [13] have analyzed the effect of thermal contact resistance between graphite and matrix on thermal property of CGI. The calculation results are close to reality when considering interfacial thermal resistance.

Creep usually takes place when  $T > 0.3T_{\rm m}(T$  is testing temperature,  $T_{\rm m}$  is melting temperature of the material) [14,15]. The creep strain of a material is described by a function of stress, temperature and time by Norton [16] and Bailey [17]firstly. Then, a  $\theta$ -projection method is proposed by Evans and Wilshire to illustrate the creep deformation [18]. Kachanov [19] and Robotnov [20] established creep constitutive equations to describe creep damage on the basis of continuous damage mechanics later. These methods are mainly summarized from empirical law but have little relationship with microstructure damage. According to work by Wu [21–23], there are four types of damages in the microstructure of nodular cast iron during thermo-mechanical fatigue test: (I) fatigue, (II) Intergranular fracture, (III) creep, (IV) oxidation. Creep interacted with fatigue is an important damages. A creep-fatigue constitutive equation is proposed on the foundation of previous researches and the calculation is close to reality. Meanwhile, the creep of

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https://doi.org/10.1016/j.msea.2018.03.039

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Received 2 January 2018; Received in revised form 8 March 2018; Accepted 9 March 2018 Available online 13 March 2018 0921-5093/ © 2018 Elsevier B.V. All rights reserved.

#### Table 1

Chemical composition of CGI alloy (mass fraction, %).

Element	С	Si	Mn	Cu	Мо	Sn	Р	S	Fe
Percentage	3.9	1.8	0.2	0.55	0.2	0.03	< 0.06	0.02-0.03	Bal.

Waspaloy nickel-base superalloy was analyzed by Wu [24]. The result suggests that dislocation glide, climb and grain boundary sliding take place during creep deformation and the creep strain is mainly caused by intragranular deformation and grain boundary sliding. The creep constitutive equation calculation is close to test results.

Although creep is an important and non-negligible damage for components at high working temperature, the available creep study is mainly focus on nickel-base superalloy and nodular cast iron. The study about CGI is mainly on strength and microstructure damage at high temperature and there is few paper reported creep behavior of CGI. Therefore, it is necessary to analyze and discern the mechanism of creep deformation and damage of CGI in order to popularize its usage and guarantee service life at high temperature. The mechanism of creep deformation and fracture of a high strength CGI is analyzed in this paper.

#### 2. Experimental materials and methods

Chemical composition of the experimental CGI is listed in Table 1. Pig iron and 45 steel were melted in GGW-0.01 medium-frequency induction furnace and poured into casting ladle at temperature 1470 °C. After being inoculated by 75FeSi and vermiculated by homemade RE-Mg-Ca respectively, the CGI melt was poured into sand mould and solidified to the wedge block, as shown in Fig. 1.

The specimens used for metallographic observation and creep test were cut in the rectangular part of the wedge block (Fig. 1ab). Microstructure of CGI was observed by Nikon-300 optical microscope. The vermicularity, pearlite and graphite content were measured by DT2000 metallographic analysis software. Creep test was conducted in RDL100 creep device, host computer was controlled by DOLI EDC222 digital measuring controller and CCPS5.0 creep test software. Temperature was adjusted by SHIMADEN FP93 instrument and the specimen deformation was measured by HEIDENHAIN SPECTO digital grating length gauge. Three N-type thermocouples were bounded in the up, middle and lower regions of the sample respectively in order to control temperature. The diagram of creep sample is shown in Fig. 2. In order to keep sample coaxial during test, an initial load (500 N, less than 10% of the test load) was applied on samples during heating. It is necessary to hold temperature stable for 1 h when temperature was up to target values (350 °C, 450 °C, 500 °C and 550 °C). Subsequently, target loads were applied on the sample (40 MPa, 100 MPa, 110 MPa, 130 MPa and 150 MPa). The testing temperature was based on the working temperature of cylinder head and the load was referred to references [25,26]. Finally, the sample after creep test was cut longitudinally in



Fig. 1. The schematic diagram of wedge-shape sample of CGI.

the gauge position by electric discharge machine and processed to metallographic specimen observed by TESCAN VEGA-II XMU scanning electron microscope (SEM). During the observation, energy dispersive spectrometer (EDS) is also used to analyze distribution of element in the corresponding composition.

#### 3. Test results

#### 3.1. Microstructure of as-cast CGI

Microstructure of as-cast CGI is shown in Fig. 3. The main microstructure of CGI is pearlite and vermicular graphite, which has strong branches and is surrounded by a few ferrite phase. The morphology of microstructure results from solidification of the alloy. On the one hand, vermicular graphite has a larger contact surface with liquid material during solidification, which means more carbon atoms can diffuse to graphite from austenite easily and quickly. Consequently, the poor carbon austenite converts to ferrite. On the other hand, the diffusion of carbon atoms is hindered by alloy elements, such as Cu, Mo and Sn, which are enriched in the boundary of austenite and have a negative effect on carbon atoms spreading to graphite from austenite. Extra carbon will transform to cementite when its solubility in austenite decreases as temperature falling down [27]. Eventually, austenite transform to pearlite. The pearlite content is 81.3% and graphite content is 8.63%. The vermicularity is 70.18%, as measured by DT2000 metallographic analysis software.

#### 3.2. Creep behavior

#### 3.2.1. Creep curve

3.2.1.1. Creep curve at different temperatures. Creep curve of CGI at different temperatures under load of 150 MPa is shown in Fig. 4. The creep strain increases with the increment of temperature. Creep strain has little variation with increment of time at 350 °C. When temperature rises to 450 °C, the creep strain increases mildly. Obvious creep deformation occurs as temperature rising to 500 °C. Creep curve rises dramatically in a short time when temperature gets to 550 °C. In a word, creep phenomenon of the CGI varies remarkable gradually with the increment of temperature.

3.2.1.2. Creep curve under different load. Creep curve of the CGI at 500 °C and 550 °C under different loads is shown in Fig. 5(a) and Fig. 5(b) respectively. The single creep curve varies with load and temperature and mainly presents two stages. At first, the curve's slope is decreasing with increment of time gradually, which suggests material hardening under load. At second, the slop keeps constant because of dynamic balance of hardening and softening. The steady creep rate increases with the increment of load and temperature. There is slight creep deformation under gentle loads and the transition from primary stage to secondary stage is not obvious. The specimen deforms severely in a short time with no obvious plastic deformation and fractures eventually when load increases from 110 MPa to 150 MPa at 550 °C.

#### 3.2.2. The creep rate of CGI

3.2.2.1. The relationship between steady creep rate and temperature. Since most remarkable creep deformation takes place under 150 MPa, the steady creep rate of CGI developing with different temperatures from 350 °C to 550 °C under 150 MPa presents in Fig. 6. It can be seen that the steady creep rate increases exponentially with temperature increment. When temperature surpasses 500 °C, steady creep rate rises dramatically and follows the exponential function:

$$\dot{\epsilon} = Aexp(BT)$$
 (1)

 $\dot{\varepsilon}$  presents steady creep rate, *T* is creep temperature, A and B are constant related to material property and load, which can be deduced by origin. Eq. (1) can be converted to another form:

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