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New aspects about reduced LCF-life time of spherical ductile cast iron due to dynamic strain aging at intermediate temperatures

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A B S T R A C T

Spherical ductile cast iron (FCD400) is widely used as container material in nuclear energy processing line due to its superior mechanical properties and low price. Fatigue properties in low cycle fatigue (LCF) can be described well by the Manson–Coffin–Basquin's rule. However, at intermediate temperature range between 453 and 723 K the elongation–temperature–diagram shows a significantly 20–10% reduced elongation and an increase in yield stress in tensile test experiments. These non-linear deviations and the phenomenon of less ductility at intermediate temperatures are known for a long time [1] [K. Chijiiwa, M. Hayashi, Mechanical properties of ductile cast iron at temperature in the region of room temperature to liquid, Imono 51 (7) (2004) 395-400]. But the following explanation is presented for the first time. In the same temperature range as the reduced fatigue life time dynamic strain ageing (DSA) also known as Portevin-le-Chartelier effect with the formation of visible serrations occurs. Both phenomena are explained by interaction effects between carbon diffusion and dislocation velocity which have at this temperature the same order of magnitude. However, this phenomenon shows interesting behavior at intermediate temperature range. During the low cycle fatigue test, DSA phenomenon disappeared, but mechanical properties show clear evidence of DSA phenomenon. Therefore, the purpose of this paper is to study the correlation of DSA occurrence, LCF and mechanical properties.

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1. Introduction

The ferritic ductile iron (FCD400) is widely used as industrial material, because it has superior mechanical properties and low price. It is also used as heat resistant material for machinery parts applied at elevated temperature. Therefore, recently, advanced mechanical properties of ductile cast iron including fatigue at high temperatures have been investigated and clarified [1–7]. In contrary to fatigue behavior of aluminium alloys [8] faster crack propagation rates due to hydrogen embrittlement can be excluded as shown in a previous paper [2]. In low cycle fatigue (LCF) of ferritic iron alloys, the fatigue life can be predicted by Coffin–Manson's model from the S–N curve by correlations of the plastic strain to the number of cycles. In this model, the ductile index is appointed as a material constant of 0.5 and the ductile coefficient is related directly to the so-called plastic deformation capacity of material. Namely, the low cycle fatigue life shall be dominated by the elongation rate. Concerned with it, one of authors reported [1] that the elongation is 20% at room temperature, but reduces to about 9% at 473 K, a temperature within the range of dynamic strain ageing (DSA), which is the phenomenon of fluctuating stress due to mobile solid solution atoms. However, the research on the DSA phenomenon is actively performed in past and present and it is confirmed that the DSA phenomenon occurred at 473 K but it cannot be confirmed at 293 K. The phenomenon of carbon and nitrogen in solid solution fixing dislocations and rearrange the structure is called DSA phenomenon. Moreover, according to the latest study [3], the elongation is 15% at 473 K compared to 20% at 293 K. Nevertheless, the experimental fatigue data can be successfully fitted with a Manson–Coffin–Basquin's equation with different parameters for intermediate temperature range between 453 and 723 K than the room temperature range [2].

So, in this study, high temperature tensile tests were carried out on ferritic cast iron at 473 K in air with the aim to clarify the effect of elongation and the occurrence of the DSA tensile phenomenon. Particularly, in this work the influence of the DSA effect on cyclic stress and elongation is studied.

2. Experiment and material

2.1. Test material

Round test pieces (diameter 30 mm, length 210 mm) of FCD400 (Japan Industrial Standard/ASTM A356) were used for the test. The

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Table 1
Chemical composition of specimen.

c	Si	Mn	P	S	Cr	Mg	Cu	Al	Sn	Fe
3.78	2.78	0.19	0.025	0.009	0.037	0.036	0.048	0.013	0.008	Balance

chemical composition of specimen and microstructure of the transverse section is shown in Table 1 and Fig. 1. The tension test and fatigue test specimens were processed as shown in Fig. 2.

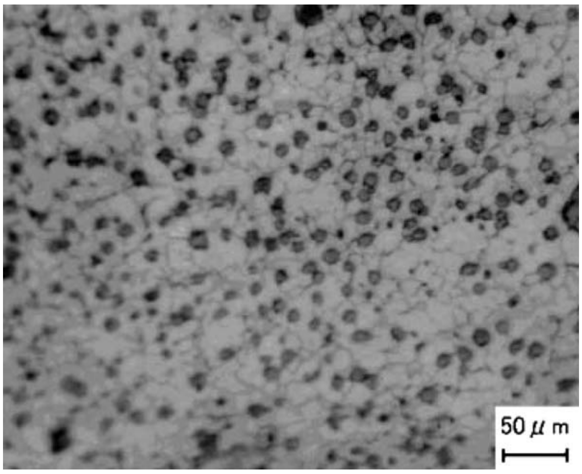

Fig. 1. Microstructure of transverse section.

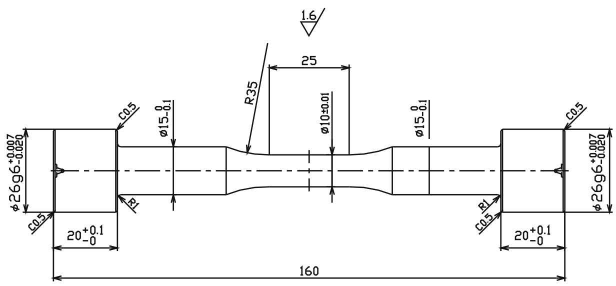

Fig. 2. Geometry of fatigue specimen.

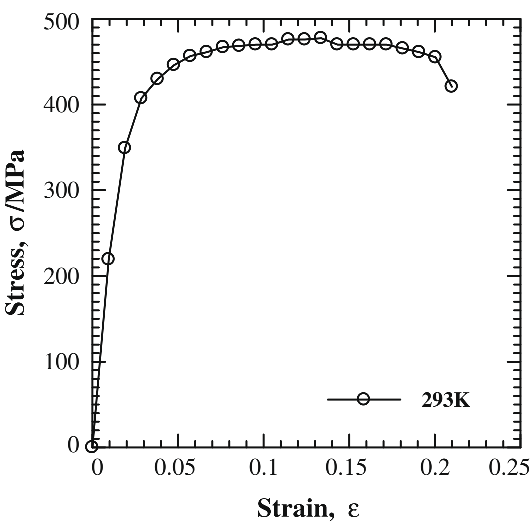

Fig. 3. Stress–strain Diagram for ASTM A356 (JIS FCD400).

Fig. 3 shows the stress–strain diagram as obtained from the tension test at 293 K as black lines, respectively. The data are also shown in Table 2. So, it was confirmed that the test pieces lie within the range of the Japan Industrial Standard (JIS) (in Table 3).

2.2. Test condition

In this study, the high temperature tensile tests and fatigue test were carried out by a computerized hydraulic servo pulsar EHF-ED100kN-TF-20L fabricated by Shimazu. The test was under taken at 473 K, at a total strain range selected from 2%, and at a constant strain rate of 0.4%/s with triangular shaped cycles applied to specimens with gage length of 15 mm and diameter of 10 mm. During test load, strain, number of repeated cycles, temperature and gauge length were detected and recorded. As described in detail elsewhere [2], the strain rate is related to ε_{tr}, the total strain range, by,

$$\dot{\varepsilon} = 2\varepsilon_{tr}/T = 2\varepsilon_{tr}f \quad (1)$$

Where T is the cycle period length of the strain, and f is the frequency (Fig. 4).

3. Experimental results and discussion

The experimental result is shown in Table 4. This time, the DSA phenomenon does not occur at 293 K (476 MPa, 21%) but occurs at 473 K (410 MPa, 9%) as confirmed by experiment. Afterwards, the DSA phenomenon in the LCF experiment occurred 30× but then it does not occur, the LCF test was able to be stopped after 162 cycles, to confirm the result that the DSA phenomenon does not

Table 2
Tensile properties on FCD400.

UTS (MPa)	0.2% Yield strength (MPa)	Elongation%	Young's modulus (Gpa)
476	382	21	227

Table 3
Test Condition: tensile Test, fatigue test.

Specimen	ASTM A356 (JIS FCD400)
Test environment	R.T. & 473 K in Air
The rate of strain (%/s)	0.4
Strain measurement	Stroke
Test environment	R.T. & 473 K in Air
Wave form of strain	Sawtooth
Total strain range ϵ_{tr} (%)	2
The rate of strain (%/s)	0.4
Strain measurement	Displacement meter (GL = 15 mm)

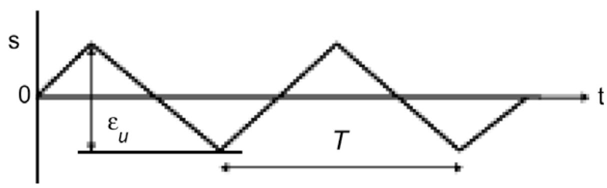

Fig. 4. Pulsating saw-toothed strain wave for strain controlled fatigue test.

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Table 4
Result of tensile test.

	UTS (MPa)	0.2% Yield Strength (MPa)	Elongation (%)	Young's Modulus (GPa)
293 (K)	476	382	21	227
473 (K)	410	328	9	44
473 (K) After fatigue test	428	342	7	61
473 (K) Annealing after fatigue test	396	317	10	40

occur in the tensile test. The result shows that the UTS is 428 MPa and Elongation is 7%. The UTS is higher and elongation is lower from virgin specimen. Moreover, when it annealed after it experimented on LCF of 162 cycles and the tensile test was done again, the DSA phenomenon was generated. The result shows UTS is 396 MPa and Elongation is 10%. The UTS is lower and elongation is higher from virgin specimen. Figs. 5 and 6 were obtained from each experimental result as shown below.

The result shown in Fig. 5 is the diagram for 'stress versus strain'. Fig. 5(a) confirms the presence of DSA and tensile tests at 473 K was performed. The result is, that at the same strain rate $\varepsilon' = 0.4\%\ s^{-1}$ as in the fatigue experiments the DSA phenomenon occurs. However, at 473 K the UTS are 410 MPa with an elongation of 8%, compared with experiments performed at 293 K, where the UTS were 476 MPa with an elongation of 21%. Hence, strength as well as elongation to fracture decreased same as in a previous study [1]. Fig. 5(b) shows that at a tensile test, which was performed by interrupting a fatigue test with total strain of 2% after 162 cycles at 473 K, DSA could not be confirmed. In this tensile test, the elongation rate until fracture was measured as 7%, a little smaller than at the tensile test at virgin specimens (8%). Moreover, the UTS increased to 428 MPa at tensile test after 162 fatigue cycles while the UTS at virgin specimens was 410 MPa. It is considered that the carbon atoms in solid solution are trapped in cores of immobile dislocations formed during the first fatigue cycles. Through this mechanism, mobile dislocations are not hindered anymore by carbon atoms, they form micro-cracks more easily and this explains the shorter LCF-life time for specimens at 473 K compared to 293 K.

This behavior was counter-checked by the following experiment, which had the intention to 'repair' such specimens, which became less ductile during fatigue at 473 K by an intermediate recovery heat treatment. A fatigue test was interrupted after 162 cycles, followed by an annealing treatment for 6 h at 723 K above

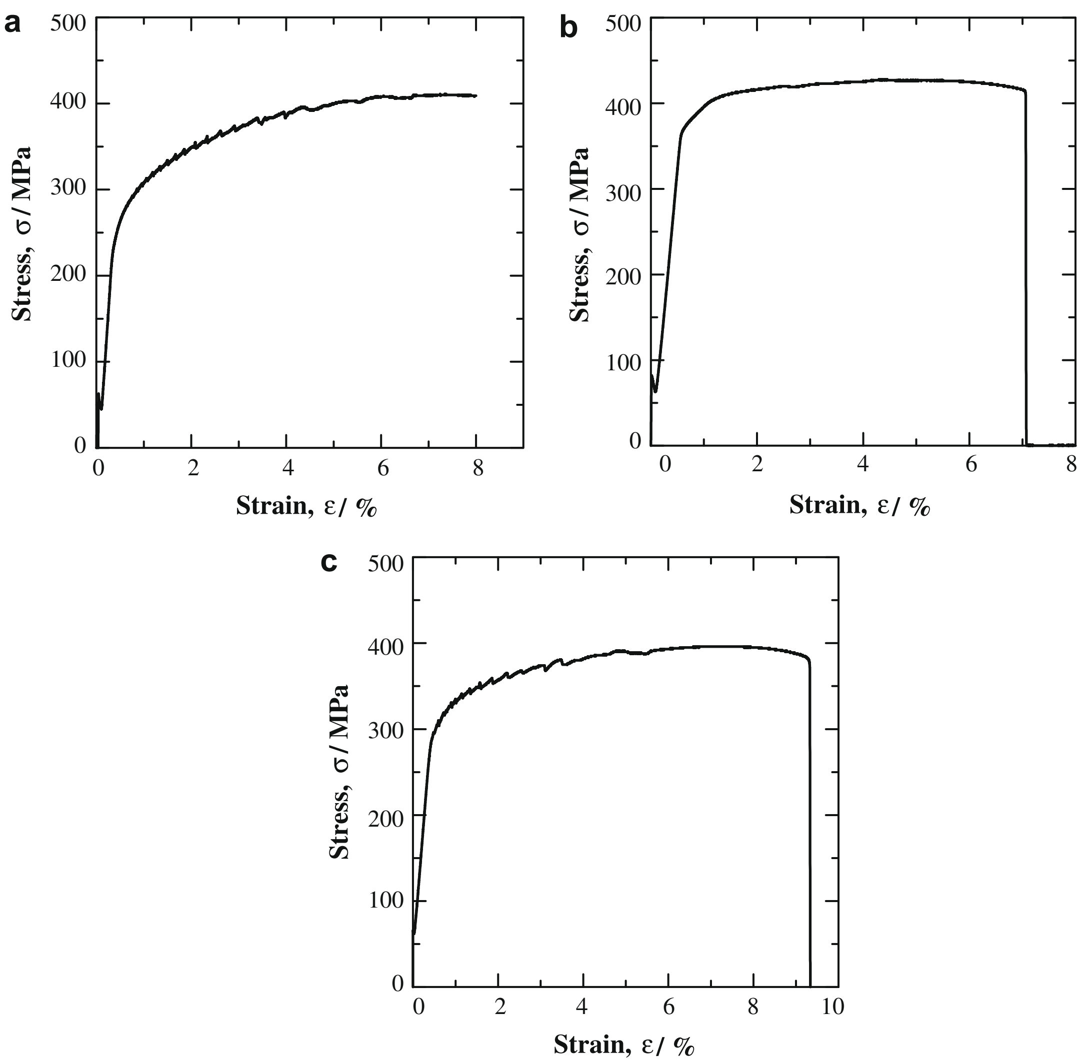

Fig. 5. Stress–strain diagrams at tensile test at 473 K for different specimens as marked: (a) Tensile test at virgin specimens, (b) Tensile test after fatigue test $N = 162$ and (c) Tensile test after fatigue test $N = 162$ and after annealing 723 K, 6 h.

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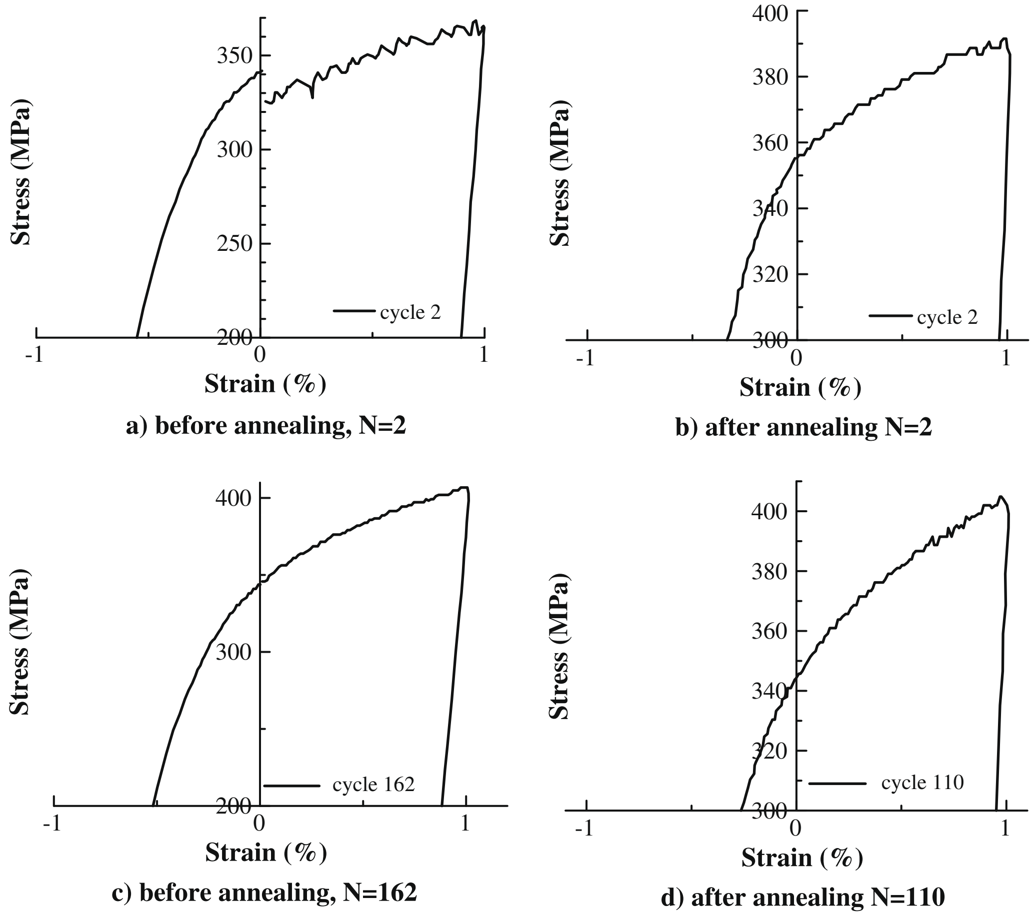

Fig. 6. Details of the fatigue hysteresis loop at specimens a, c) before b, d) after annealing 723 K 6 h, a, b) after two cycles, c), d) a few cycles later.

the recrystallization temperature. Indeed in a subsequent tensile test the DSA serrations were conformed and the fracture elongation increased to 10%, but the UTS decreased further to 396 MPa. It is considered that, during the short fatigue period obviously some dislocations continued to form micro-cracks, which causes the remarkable low tensile strength compared to the virgin specimens and the in total shorter life time ($N_f = 404$ in the case of virgin specimens compared to $N_f = 162 + 113 = 275$ cycles in case of the recovery treatment).

Another goal of this experiment was to check, whether the DSA effect appears again after the recovery annealing, or in other words whether the mobility of the carbon atoms could be retained in order to achieve the Cottrell cloud hardening mechanism.

Table 5 shows the results of low cycle fatigue (LCF) tests. At 473 K the virgin specimen has a life time of 404 cycles. However, if we focus on the plastic strain of the virgin specimen, which is 0.56, 0.53 after 162 cycles and is 0.54 after annealing, in other words in all three cases it has nearly the same number, but the life time decreases down to 129 cycles.

Fig. 6 shows enlarged parts of the corresponding hysteresis loops as generated from the fatigue test data. At a total strain of 2% the maximum and minimum stresses after two cycles at the specimens before and after annealing are almost unchanged, confirming saturation in the stress level. The magnified parts confirmed that the DSA phenomena occurs at $N = 2$ before and after annealing (Fig. 6(a) and (b)), but a few cycles later it disappears Fig. 6(c). Nevertheless, cycle 110 still have DSA phenomenon Fig. 6(d). The recovery experiment could confirm, that the DSA is retained, which means dislocation are annihilated and carbon atoms can move freely, but a few cycles later the dislocation trapping works and DSA disappears. Hence, this experiment was successful to explain DSA and indeed the dislocation fixing by Cottrell clouds is recovered partially, but concerning the other purpose of finding a method to retain the high strength, it failed and probably requires the application of compressive stress during recovery in order to close the formed micro-cracks.

As a summary, the characteristic occurrence of tensile behavior seen in the experimental data can be explained by the dynamic

Table 5
Result of thermal low cycle fatigue test.

	Total Strain (%)	Elastic strain	Plastic strain	Stress (MPa)			Cycle
				Max	Min	Range	
473 (K)	2	1.4309	0.5691	633	−637	1270	404
473 (K) Fatigue test before annealing		1.466	0.534	407	−435	842	162
473 (K) Annealing after fatigue test		1.4537	0.5463	407	−431	838	113

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strain ageing. A complete solid solution hardening works from 293 to 400 K according to literature data [1], because the dislocation velocities are faster than the diffusion of the carbon atoms. Between 400 and 473 K the dynamic strain aging occurs at low stress levels, due to the solid solution of the carbon atoms which rearrange immediately according to the dislocation movement. In other words, DSA happens because of the competition of almost the same velocities of both, the dislocations and the diffusion of the carbon atoms. This was deduced from tensile tests on cast iron in the range of 423–573 K [2,3]. At high stress levels, DSA does not occur, because the number of mobile dislocations is too few or the dislocation velocities are so high, that the trapped carbon atoms cannot contribute to any hardening effect. At higher temperatures (>573 K) the solid solution hardening mechanism works perfect, because the carbon diffusion is faster than the dislocations.

4. Conclusion

In this research, thermal tensile tests were performed on ferrite ductile cast iron at elevated temperatures (473 K) under ambient atmosphere. The obtained results are as following.

The UTS at 473 K of virgin specimen is higher (428 MPa) compared to fatigued specimens at 473 K (410 MPa) or specimens after an attempted recovery treatment (723 K 6 h, 396 MPa), where only the elongation slightly increased.

In the tensile tests at 473 K the DSA phenomena occurs regardless of the height of the strain, but at fatigue tests DSA occurs only at the first few initial cycles, in later cycles carbon is trapped and DSA disappears.

Tensile test behavior of ductile cast iron can be distinguished in three different temperature ranges (293–400 K, 400–573 K, >573 K) according to the competition of dislocation speed and diffusion speed of carbon atoms. In the middle range, where both velocities are compatible, dynamic strain aging (DSA) occurs.

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