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STUDY ON THE EVALUATION METHOD FOR SUPERPLASTICITY OF DUPLEX STAINLESS STEEL

ŠTUDIJ EVALUACIJSKE METODE ZA SUPER-PLASTIČNO DUPLEKS NERJAVNO JEKLO

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Aiming at the problem of long duration of superplastic tensile tests for materials with a large elongation, a new method for evaluating the superplastic properties of duplex stainless steel (DSS) is proposed. In this paper, relative values of stress difference D and stress variation factor f are introduced to predict the elongation, δ. Elongation can be predicted with the relational expression between D and δ as long as the specimen is stretched only by 500 % during the superplastic uniaxial tensile test. Factors affecting elongation include the thermo-mechanical treatment and superplastic-deformation conditions. For DSS2205, the change trend of f and influencing factors is similar to that of elongation and influencing factors.

Keywords: duplex stainless steel, superplastic tension, evaluation method, stress variation factor

Avtorji prispevka so, glede na težave z dolgotrajnim preizkušanjem super-plastičnih materialov pod natezno obremenitvijo, predlagali novo metodo za ovrednotenje super-plastičnih dupleks nerjavnih jekel (DSS). V tem članku uvajajo relativne vrednosti napetostne diference D in faktor variiranja napetosti f za napoved raztezka δ. Raztezek se lahko napoveduje z izrazom med D in δ tako dolgo dokler se raztezek preizkušanca ne poveča za 500 % med dolgotrajnim super-plastičnim enoosnim nateznim preizkusom. Faktorja, ki vplivata na raztezek sta termomehanska obdelava in pogoji super-plastične deformacije. Pri jeklu DSS 2205 je sprememba trenda f podobna trendu raztezka in vplivnim faktorjem.

Ključne besede: dupleks nerjavno jeklo, super-plastično natezanje, evaluacijska metoda, faktor variiranja napetosti

1 INTRODUCTION

Superplasticity has been formally defined as the capability of a fine-grained polycrystalline solid to exhibit a notable ductility over hundreds of percent of elongation at a high temperature and/or low strain rate.[1,2] The high ductility achieved through superplastic materials bears industrial significance as it forms the basis for fabrication methods for producing complex shapes. The main parameters to evaluate superplasticity include elongation to failure, strain-rate sensitivity index, deformation homogeneity and activation energy.[3–5] Among these parameters, elongation and strain-rate sensitivity index, m, are the most important.[4]

In general, elongation shows a monotonic increase with an increasing m-value.[6–10] However, several papers[11–13] concerned with superplastic materials reported that the peak ductility does not necessarily occur at maximum m. G. Rai and N. J. Grant [11] elaborated that the maximum value of m ($\approx$0.9) occurs at a strain rate of 10^{-3} s^{-1} whereas elongation to fracture totals 700 %, in contrast to 1200 % obtained at a lower strain rate when the initial m is $\approx$0.08. H. Miyamoto et al.[12] showed that the total elongation of Fe-25Cr-7Ni-3Mo-0.14Ni reaches the maximum at an intermediate strain rate, whereas the m-value increases monotonously with an increasing strain rate. H. Zhang[13] concluded that "higher *m*-values alone do not necessarily lead to higher elongations." For different materials, errors in the monotonic increase of the m-value with the increasing elongation often occur. In ultrahigh-strength steels, martensite and carbides may cause internal-stress concentrations or cavities during a deformation, thus limiting the superplastic elongation at high *m*-values.[13] For example, the average m-value of 1.3CMnSiCr steels of AC (air cooled) samples equals 0.44 at 1023 K, whereas the average elongation amounts to $\approx$233 %.[13]

However, duplex stainless steel (DSS) exhibits excellent superplasticity, and an elongation of more than 1000 % can be obtained under certain circumstances[14–17] at a low m-value. The maximum elongation value of 25Cr-6.5Ni-3Mo-0.14N[17] goes above 2000 %, whereas the calculated m-value is č0.46. The peak elongation of Fe-24Cr-7Ni-3Mo-0.14N[18] of 750 % is obtained, whereas the strain-rate sensitivity is $\approx$0.37. Other researchers pointed out that m is strain dependent, thus a function of strain.[19,20] A. K. Ghosh[19] suggested that the terminal *m*, i.e., *m* near fracture, not the initial yield, would be expected to control the elongation at fracture. In

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addition, the measurements of this parameter are challenging, and varied results are observed from different test procedures.[10] Thereby, the m-value alone proves insufficient for evaluating the superplastic behaviour.

Elongation is one of the most commonly used measures for characterizing superplastic properties. As most of the superplastic uniaxial-tension experiments are conducted at a constant crosshead speed, the test time is notably long for the materials with a high elongation. For a tensile specimen with a gage length of 10 mm, the test time is 10^4 s when the initial strain rate is 10^{-3} s^{-1}, and the elongation is 1000 %; it reaches 10^5 s when the strain rate is 10^{-4} s^{-1}. In addition, only the difference in the gage dimension results in a different elongation. Taking a Zn-22%Al alloy as an example, after the same treatment, the elongation of the alloy with a gage diameter of 5.994 mm (≈400 %) becomes much higher than that with a 0.1524-mm gage diameter (≈30 %) under the same deformation condition.[21] In the empirical sense, differences in the material preparation, test machine state, operation level and control capability influence superplastic elongation. Under the same deformation conditions, superplastic elongation still varies substantially. Therefore, evaluation of the superplastic properties of materials based on elongation is subject to certain limitations.

In view of the abovementioned problems, the parameter that can reduce the time consumed and efforts devoted and enhance the accuracy and completeness needed for predicting the superplastic property of a given metal alloy is still required. For this purpose, our paper proposes relative values of stress difference at two points after the peak strain, *D*, to evaluate the superplasticity. The reciprocal of *D*, the stress variation factor, *f*, is introduced. In addition, the relationships between *f* and elongations under different thermo-mechanical treatments and deformation conditions of DSS2205 are discussed. In this work, the stress–strain curve of the superplastic uniaxial tension with a fixed displacement rate is explored to determine *D* and *f* under different conditions. Elongation can be predicted based on *f* or *D* by stretching the samples only to a given elongation of 500 %, which greatly shortens the experimental time.

2 EXPERIMENTAL PART

Table 1 lists the types and chemical composition of DSSs used in this study. Among these DSSs, 2205 and 2906 are used as experimental steels by the author of this paper. S1, S2, D1 and D2 were used by the Japanese scholar Y. Maehara.[22] Industrially cast, hot-rolled and solution-treated strips were laboratory cold-rolled to 2 mm. To examine the effect of the thermo-mechanical-treatment conditions on superplasticity, the solution treatment temperature and cold-rolled reduction ratio were varied. The as-received hot-rolled steel was solution treated at a temperature range of 1050–1350 °C for 30 min, followed by water quenching. The hot-rolled steel of different thicknesses was then cold rolled to 2-mm sheets with different reduction ratios ranging from 0 % to 85 %. Dog-bone-shaped tension specimens with a gage length of 10 mm, width of 6 mm and thickness of 2 mm were cut parallel to the rolling direction, using spark cutting, from the cold-rolled sheets. The specimens were strained isothermally at a constant crosshead speed. Tensile tests were carried out in a temperature interval of 850–1000 °C, at strain rates ranging from $5\times10^{-}$ s^{-1} to 1.5×10^{-3} s^{-1}. After reaching the selected testing temperature, the specimens were homogenized for (1, 2, 3, 5, 7, 10, 15, 20 and 30) min prior to applying the load in order to study the effect of the holding time on the superplasticity.

3 RESULTS AND DISCUSSION

3.1 Stress–strain curves of DSS

The stress–strain curve for superplastic axial tension generally involves two forms, i.e., the true stress–strain curve and the engineering stress–strain curve. **Figure 1** shows the two kinds of stress–strain curves for the superplastic tension of DSS2205 at a solution temperature of 1200 °C, deformation temperature of 950 °C, strain rate of 1.5×10^{-3} s^{-1} and cold-rolling reduction ranging from 0 % to 85 %. In the stress–strain curve, after the peak stress, the flow stress decreases until the final fracture. The inflection point mathematically refers to the point that changes the curve in the upward or downward direction. Intuitively speaking, the inflection point is the concave–convex boundary point of the curve. As observed on **Figure 1**, the largest difference between the two kinds of curves is the fact that the engineering stress–strain curves show the inflection point after the

Table 1: Types and chemical compositions of DSSs, (*w*/%)

Steel	Cr	Ni	Mo	N	C	Si	Mn	Cu	P	S	W	Fe
2205	22.05	5.37	3.22	0.15	0.017	0.42	1.10	0.043	0.024	0.006	–	Bal
2906	30.06	7.26	2.86	0.47	0.0064	0.25	0.53	0.52	–	–	–	Bal
S1	18.52	4.06	–	0.0094	0.0015	1.69	3.22	1.18	0.005	0.002	–	Bal
S2	18.77	3.96	–	0.00928	0.0015	1.73	3.42	1.20	0.005	0.002	–	Bal
D1	24.66	6.82	2.79	0.143	0.0017	0.48	0.85	0.46	0.025	0.001	0.28	Bal
D2	22.88	10.52	–	0.0912	0.0005	3.22	0.79	–	0.003	0.001	–	Bal

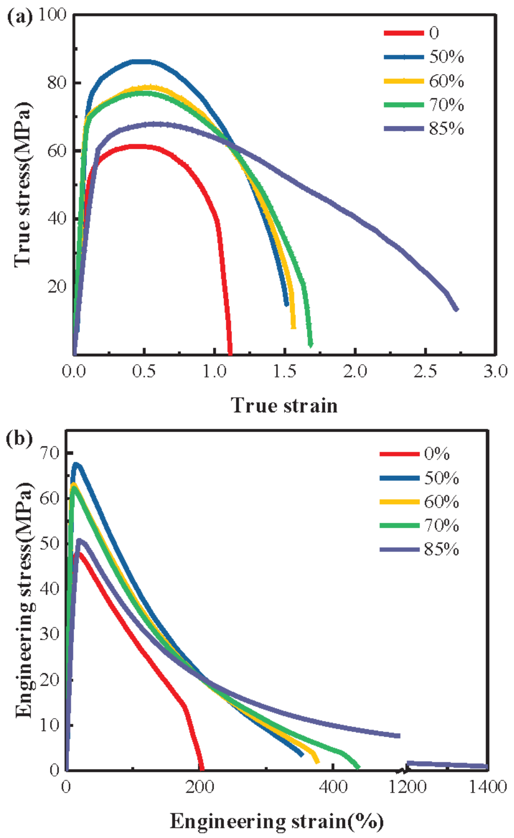

Figure 1: Stress–strain curves of DSS2205 during superplastic uniaxial tension: a) true stress–strain curve, b) engineering stress–strain curve

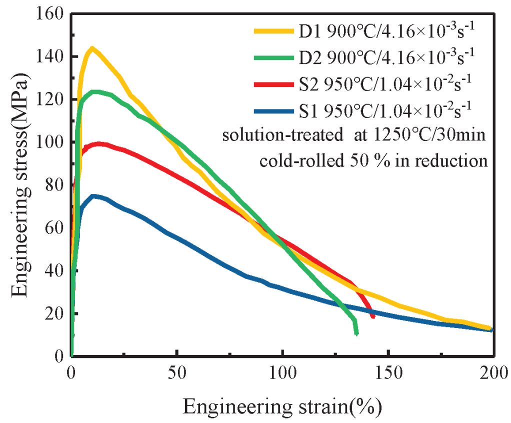

Figure 2: Engineering stress–strain curves of four DDSs (original data of DDS superplastic materials from Y. Maehara[22])

peak strain, whereas the true stress-strain curves exhibit otherwise. This finding coincides with the uniaxial tension of most superplastic DSSs; however, an inflection point also exists after the peak strain in certain true stress–strain curves.[23]

Experience has shown that if an inflection point occurs on the stress–strain curve, the flow stress after the peak stress decreases slowly; otherwise, it drops rapidly. For a material without an inflection point on the engineering stress–strain curve, elongation is usually less than 200 %. **Figure 2** shows examples of flow stress–strain curves of various steels cited in [22]. **Figure 2** shows the absence of inflection points on the curves of S2 and D2 and their presence on the curves of S1 and D1. After passing the peak stress, the flow stress decreases gradually until the final fracture of steels S1 and D1. However, a sudden stress decrease corresponding to the local necking is observed in steels S2 and D2. S1 and S2 exhibit poor ductility and can be regarded as non-superplastic materials under this condition. It can be used to assess whether a DSS is a superplastic material according to the inflection point after the peak strain on an engineering stress–strain curve; that is, if an inflection point exists on the curve, the DSS is superplastic; otherwise it can be regarded as a conventional one.

Figures 1 and **2** reveal that the stress after the inflection point decreases more slowly with a higher elongation than with a lower elongation. To describe the change rule of stress with strain more intuitively, the first derivative $d\sigma/d\varepsilon$ is obtained for the stress–strain curve of **Figure 1**, and the obtained result is shown in **Figure 3**. The peak point appears where $d\sigma/d\varepsilon = 0$ and the inflection point appears where $d^2\sigma/d\varepsilon^2 = 0$, i.e., the first derivative is the extreme value.

Figure 3a illustrates that for the true stress–strain curve, the first derivative $d\sigma/d\varepsilon$ decreases with the

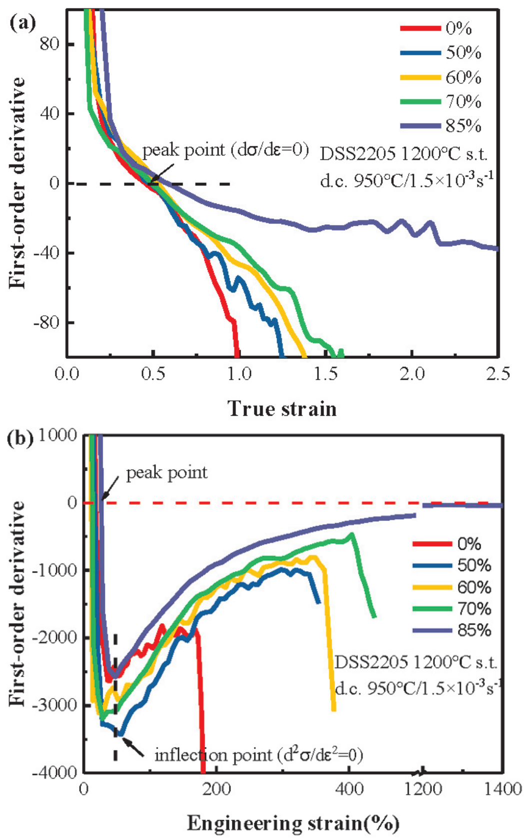

Figure 3: First-order derivative diagrams of stress–strain curves corresponding to **Figure 1** (note: s.t. stands for solution treatment; d.c. stands for deformation condition; holding time is 5 min unless otherwise stated; the same below)

increasing strain, whereas the slope of the curve varies notably under different conditions. When the elongation is higher, the curve after the peak strain decreases more slowly, indicating a better superplasticity of the material. After the peak strain, the discrimination degree of the dσ/dε-ε curve under different cold-rolling reduction ratios becomes more notable with the increasing strain. This condition provides the possibility to evaluate the superplasticity of the material using the first derivative dσ/dε at a certain strain after the peak strain. Given the lack of inflection point for the true stress–strain curve used, no extreme value exists on the first derivative curve.

For the inflection point in the engineering stress–strain curve corresponding to **Figure 1b**, an extreme value exists in the first derivative diagram shown in **Figure 3b**. The first derivative value first decreases with the strain and then increases, whereas the first derivative at the inflection point has its minimum value. A larger first derivative (i.e., a smaller absolute value) indicates a higher elongation. After the inflection-point strain, the discrimination degree of the dσ/dε-ε curve under different cold-rolling reduction conditions also becomes notable.

Owing to uncertain factors during superplastic uniaxial stretching, elongations differ under the same conditions as shown in **Figure 4**. **Figure 4a** depicts the two engineering stress–strain curves of DSS2205 deformed at 950 °C and an the initial strain rate of 1.5 × 10^{-3} s^{-1} with the solution at 1200 °C and a cold-rolled reduction ratio of 85 %; **Figure 4b** depicts the two engineering stress–strain curves of DSS2205 deformed at 950 °C and the initial strain rate of 7.5 × 10^{-4} s^{-1} with the solution at 1200 °C and a cold-rolled reduction ratio of 85 %. As observed from **Figure 4**, the elongations differ under the same thermo-mechanical treatment and deformation conditions, but the slope of the stress–strain curve dσ/dε is roughly the same between the inflection-point strain and 400 %. When the strain is higher than 400–500 %, the slope of the curve begins to change. In addition, for the samples with a high elongation, the peak stress is high, which may be related to the difference in the length of the uniform deformation zone; that is, a longer length of the uniform deformation zone denotes a higher superplastic elongation.

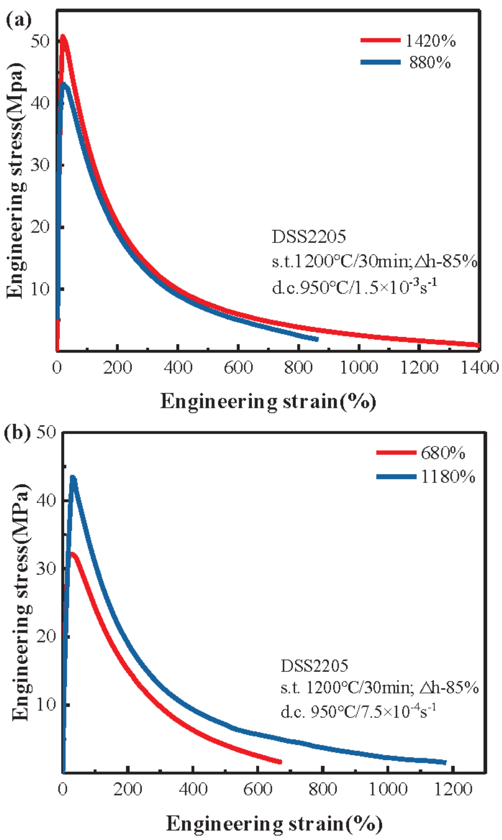

Figure 4: Engineering stress–strain curves with different elongations of DSS2205 under the same precondition and deformation condition (note: Δh represents the cold-rolling reduction ratio; the same below)

3.2 Evaluation of superplasticity with the stress difference

Theoretically, the superplasticity of a material can be evaluated using dσ/dε at a certain strain after the peak strain. However, in practical applications, the smoothness degree of stress–strain curves must be particularly high when obtaining the dσ/dε-ε curve. Numerous factors affect the curve vibration during a tension test. Excessively artificial smoothing distorts the curve, thus increasing the accumulative error of dσ/dε. Therefore, this paper proposes to evaluate the superplasticity by adopting a relative value of the stress difference, *D*, which can lower the standards for the smoothness of a curve and reduce the error caused by curve fluctuation.

The Backofen equation is the most commonly used constitutive equation for describing superplastic flow characteristics:[16]

$$\sigma = K\dot{\varepsilon}^{m} \tag{1}$$

where σ refers to the superplastic flow stress; $\dot{\varepsilon}$ denotes the strain rate; m represents the strain-rate sensitivity index; and K is a material constant.

For the superplastic tension with a constant strain rate, the tensile speed (i.e., tester chuck speed) increases with the increasing strain. This condition requires precise control of the tensile speed of the testing machine, which is slightly more complex than the one under a constant tensile speed. Therefore, most of the superplastic tensile tests are carried out under constant tensile-speed conditions, and the strain rate decreases gradually during a deformation.

According to the plasticity theory, the relation between the strain rate and the tensile speed under a constant tensile speed is given by the following Equation (2):

$$\dot{\varepsilon} = \frac{\dot{u}_0}{l_0(1+\varepsilon)} = \frac{\dot{\varepsilon}_0}{1+\varepsilon} \tag{2}$$

where ε corresponds to the strain; $\dot{\varepsilon}_0$ stands for the initial strain rate; l_0 signifies the original length of gage segment; and $\dot{u}_0$ is the tensile rate.

Substituting Equation (2) into Equation (1) and Equation (3) becomes obtainable:

$$\sigma = K\left(\frac{\dot{\varepsilon}_0}{1+\varepsilon}\right)^m \tag{3}$$

In the engineering stress–strain curve, two points (A and B) after the peak stress are selected: σ_A and ε_A, σ_B and ε_B are set to the engineering stress and engineering strain of points A and B, respectively. When the strain difference between points A and B is small, the m-value is assumed to change minimally within this range. According to **Equation 3**, the relative values of the stress difference between σ_A and σ_B, that is $D_{(A,B)}$, can be expressed by **Equation 4**:

$$D_{(A,B)} = \frac{\sigma_A - \sigma_B}{\sigma_A} = 1 - \left(\frac{1+\varepsilon_A}{1+\varepsilon_B}\right)^m \tag{4}$$

The stress–strain curves show that a smaller relative value of stress difference results in a higher superplastic elongation of the material. **Equation 4** indicates that a larger m implies a larger relative value of the allowable stress difference and vice versa.

The discrimination degree of the dσ/dε–ε curve under different conditions becomes more notable with the increasing strain (**Figure 3**). To reduce the test time, the selected cut-off strain is as small as possible. Thus, the most formable strain range must be determined to accurately characterize the superplasticity.

Considering the peak strain ε_p as the strain zero point, two points (A, B) of the strain difference are selected. For example, in the engineering strain–stress curve, ε_p = 30 %, ε_A = 60 % and ε_B = 110 %. Then, under the above assumptions, ε_p = 0 %, ε_A = 30 % and ε_B = 80 %. The corresponding $D_{(A,B)}$-value can be obtained by analysing the stress–strain curves. In order to test and verify the feasibility of the proposed parameter, the relationship between δ under various conditions mentioned in the experimental part and $D_{(A,B)}$ corresponding to different strains was investigated. It is particularly important to choose the strain values of A and B. **Figures 5a**, **5b**, **5c**

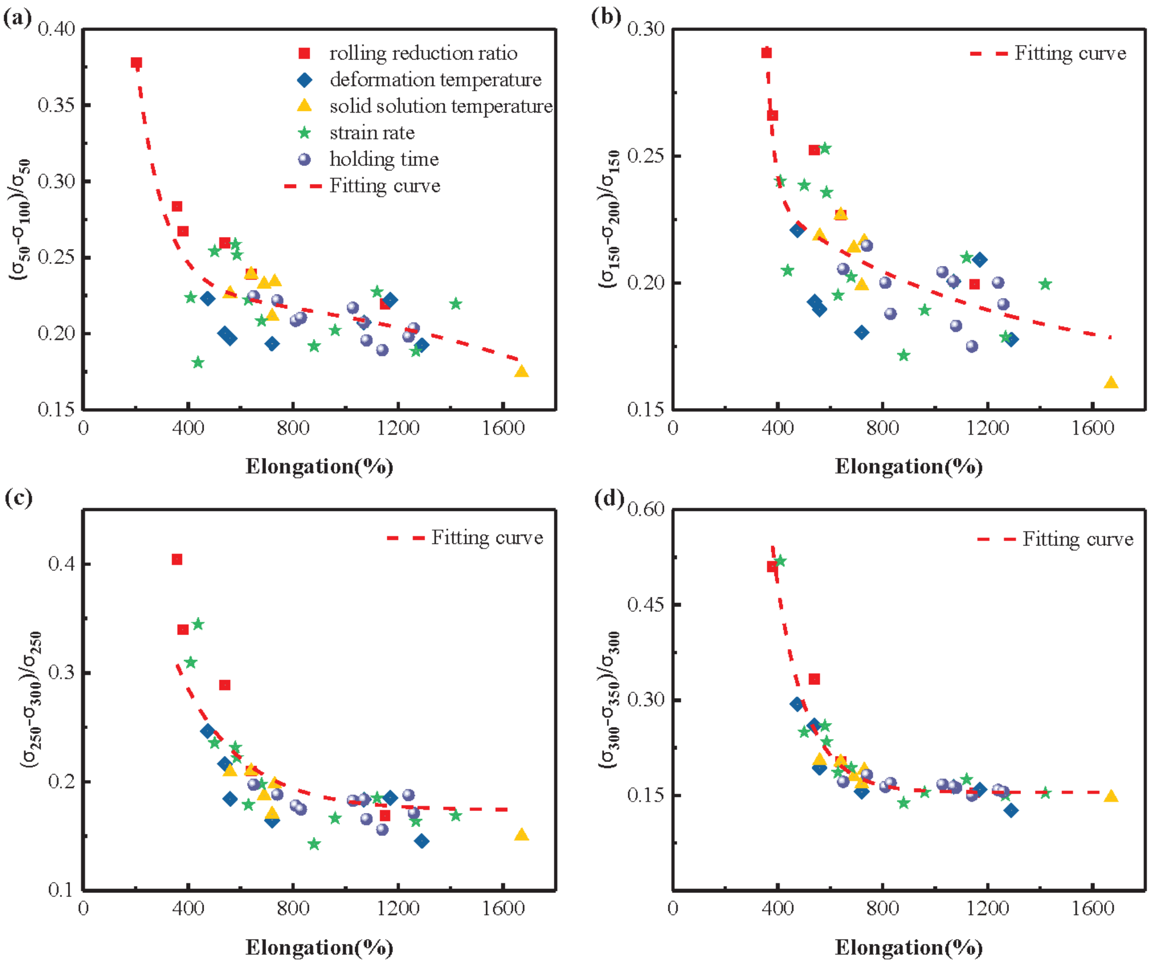

Figure 5: Relationships between relative values of the engineering-stress difference and elongation of DSS2205: a) ε_A = 50 %, ε_B = 100 %, b) ε_A = 150 %, ε_B = 200 %, c) ε_A = 250 %, ε_B = 300 %, d) ε_A = 300 %, ε_B = 350 %

and **5d** show the relationships between δ and $D_{(50,100)}$, $D_{(150,200)}$, $D_{(250,300)}$, and $D_{(300,350)}$, respectively. When the selected strains of A and B are larger, the relationship between δ and $D_{(A,B)}$ is more relevant. When ε_A = 300 % and ε_B = 350 %, there is a good correspondence between δ and $D_{(A,B)}$. To teremine the relative difference between the engineering stress and true stress, the superscript e is added, that is, $D^e_{(A,B)}$. The relationship can be expressed with Equation (5).

$$D^e_{(300,350)} = \frac{\sigma_{300} - \sigma_{350}}{\sigma_{300}} = \\ = 5.526\exp\left(-\frac{\delta}{116.745}\right) + 4.53\exp\left(-\frac{\delta}{116.75}\right) + 0.155 \tag{5}$$

In general, the peak strain of the engineering stress–strain curve of DSSs is less than 50 %. When ε_A = 300 % and ε_B = 350 %, the total cut-off strain reaches less than 500 %. Therefore, the superplastic tension with a constant tensile speed can be stretched only to 500 % and elongation is predicted in accordance with **Equation 5**, whereby the test time can be remarkably shortened.

The peak strain in the true stress–strain curve is generally greater than the one in the engineering stress–strain curve. For example, the peak true strain of DSS2205 studied in this paper generally appears at around 100 %. By adopting the method mentioned above and analysing the relationship between the relative values of the true-stress difference and elongation when ε_A = 50 % and ε_B = 100 %, after the peak true strain is selected, a good correspondence between them is achieved (**Figure 6**). The relationship can be expressed with Equation (6).

$$D^e_{(50,100)} = \frac{\sigma_{50} - \sigma_{100}}{\sigma_{50}} = \\ = 2698.165\exp\left(-\frac{\delta}{18.062}\right) + 0.323\exp\left(-\frac{\delta}{225.49}\right) + 0.034 \tag{6}$$

Figure 7 shows the relationship between the relative values of the engineering stress difference and elongation of DSS2906 at ε_A = 300 % and ε_B = 350 %. The corresponding fitting formula can be expressed with Equation (7):

$$D^e_{(300,350)} = \frac{\sigma_{300} - \sigma_{350}}{\sigma_{300}} = \\ = 6.081\exp\left(-\frac{\delta}{8.576}\right) + 1.193\exp\left(-\frac{\delta}{203.8}\right) + 0.121 \tag{7}$$

Comparing Equations (5), (6), and (7), the forms of the fitting formulas are the same, but the corresponding coefficients differ. To determine these coefficients, several sets of elongation values and corresponding stress differences are needed, and then obtained with the nonlinear-curve-fitting method in Origin Software.

3.3 Evaluation of superplasticity with stress-variation factors

As $D_{(A,B)}$ decreases with the increasing δ, $1/D_{(A,B)}$ is proposed for the evaluation of superplasticity. Furthermore, in the following description, $\sigma_{300}/(\sigma_{300}\text{-}\sigma_{350})$ is referred to as the stress-variation factor and it is represented by f. **Figure 8** shows the variation rule of the stress-variation factor and measured elongation of DSS2205 with the solution temperature, cold-rolling reduction, deformation temperature, strain rate and holding time. With an increase in the deformation temperature, δ and f increase when deformed at a strain rate of 1.5×10^{-3} s^{-1} with a solution temperature of 1300 °C and a rolling reduction of 85 % as seen on **Figure 8a**. **Figure 8b** shows that δ and f of DSS2205 with an 80-% rolling reduction first decrease and then increase with an increase in the solution temperature when tested at 950 °C with a strain rate of 1.5×10^{-3} s^{-1}, and the minimum value is obtained at 1150 °C. **Figure 8c** shows that the relationships between δ, f and strain rate are parabolic when the sample is deformed at 950 °C, with a solution temperature of 1200 °C and rolling reduction of 85 %. **Figure 8d** indicates that that the relationships between δ, f and holding time are wavy when the sample

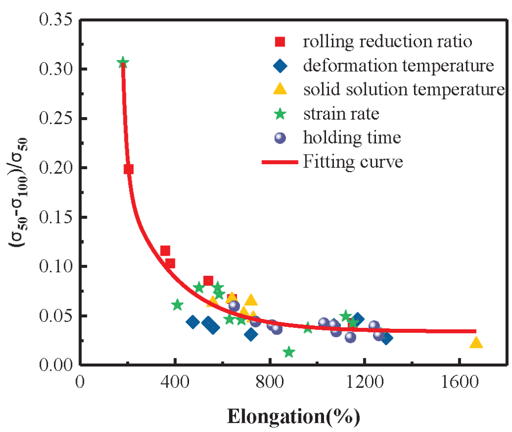

Figure 6: Relationship between relative values of the true-stress difference and elongation of DSS2205

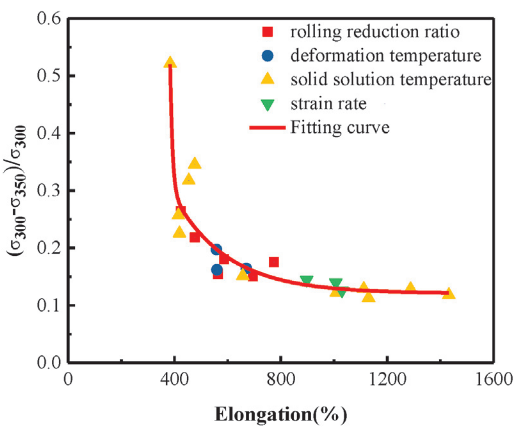

Figure 7: Relationship between relative values of the engineering-stress difference and elongation of DSS2906

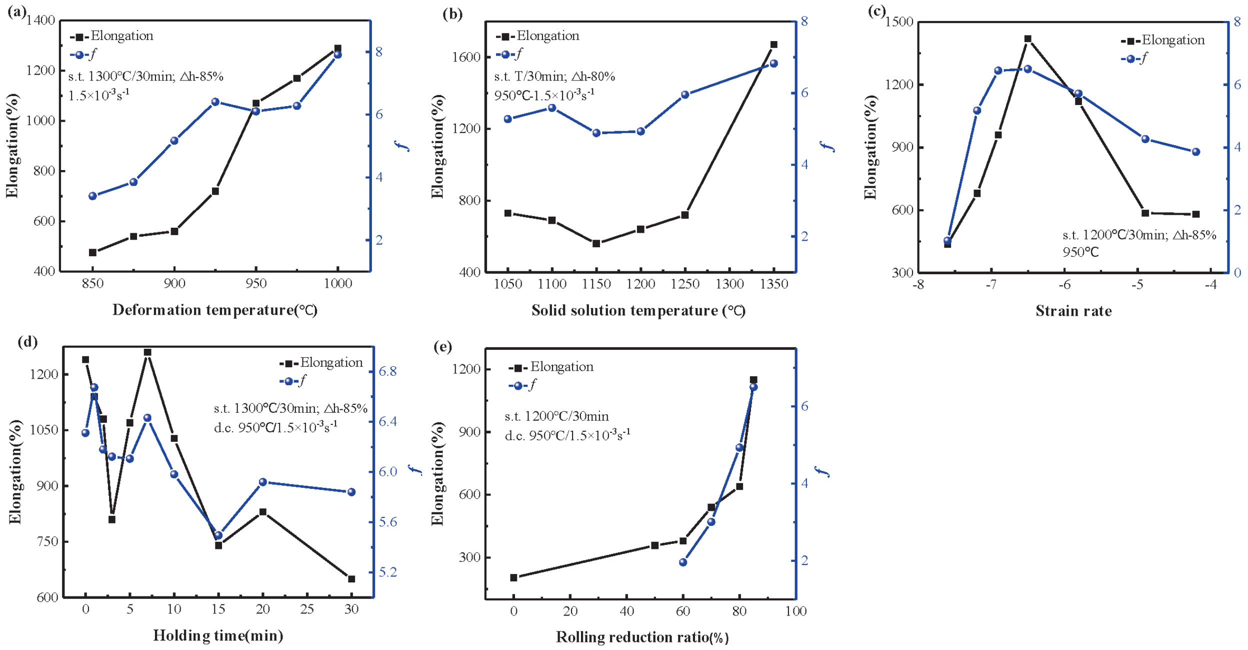

Figure 8: Variation rule for *f* and *δ* with different pre-treatment and deformation conditions for DSS2205

is deformed at 950 °C, with a strain rate of 1.5×10^{-3} s^{-1}, solution temperature of 1300 °C and rolling reduction of 85 %. **Figure 8e** demonstrates that *δ* and *f* increase with an increase in the rolling reduction when the sample is deformed at 950 °C, with a strain rate of 1.5×10^{-3} s^{-1} and solution temperature of 1200 °C. As depicted in **Figure 8**, the trends of *f* and *δ*, as a whole, are almost the same under pre-treatment and superplastic-deformation conditions. Therefore, *f* can be used to predict the change rule of elongation under specific deformation conditions.

4 CONCLUSIONS

The stress–strain curves of DSS2205 and DSS2906 during a superplastic deformation are investigated. The main conclusions of the present study include the following:

- Whether a material is superplastic can be assessed with respect to whether an inflection point occurs after the peak strain of the engineering stress–strain curve; that is, a superplastic material has an inflection point and a conventional one is without an inflection point.
- When applying superplastic uniaxial tension with a constant tensile speed and the sample is stretched to 500 %, the material elongation can be predicted in accordance with the change rule of $D^{e}_{(300,350)} - \delta$. This condition may lead to a significant reduction in the total experiment time for a material with a high elongation.
- For DSS2205, the stress-variation factor and elongation show a good consistency with the changes in the thermo-mechanical treatment and superplastic-deformation conditions.

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