Rev.Adv.Mater.Sci. 10 (2005) 171-175

MECHANICAL AND CREEP PROPERTIES OF ELECTRODEPOSITED NICKEL AND ITS PARTICLE-REINFORCED NANOCOMPOSITE

V. Sklenicka[1], K. Kucharova[1], M. Pahutova[1], G. Vidrich[2], M. Svoboda[1] and H. Ferkel[2]

[1] Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Zizkova 22, CZ-616 62 Brno, Czech Republic

[2] Institute of Materials Science and Technology, Clausthal University of Technology, Agricolastrasse 6, D-38678 Clausthal-Zellerfeld, Germany

Received: April 22, 2005

Abstract. A comparison between the tensile properties and the creep characteristics of the electrodeposited unreinforced nickel and its nanocomposite reinforced by 2 vol.% of nano-sized SiO_2 particles at temperatures in the range from 293 to 473K shows that the tensile properties of the composite are not considerably improved compared to those of the matrix nickel. By contrast, the presence of the particle reinforcement may lead to an increase of the creep resistance of the composite.

1. INTRODUCTION

One major problem with research on the mechanical and/or creep properties of the nanocrystalline materials (nc-materials) is obtaining bulk fully-densified material. The method which has been commonly employed to synthesise coating nc-materials is an electrodeposition [1,2]. It is a simple, inexpensive, and versatile method to produce dense metallic nanocrystals. Further, an electrolytic co-deposition is a well-known and suitable process for the production of particle compound materials (discontinuous particle nanocomposites) [3].

Another problem is the lack of understanding of the deformation and fracture mechanisms and the thermal stability of microstructure of nanocrystalline materials. Microstructural and dimensional constraints at the nano-scale may break down or even reverse conventional size laws in materials behaviour [4]. Although extensive information is available on the mechanical and/or creep properties of conventional polycrystalline metallic materials the research work on nanocrystalline materials has been considerably limited. Nano-nickel (Ni-nc) is one of the nanocrystalline metals that have been subjected to systematic scientific investigations in the areas of thermal stability [5-7] and mechanical properties [8-12] and/or creep [8,13].

The present paper describes the result obtained in an investigation that was undertaken to explore the potential for introducing significant strengthening into a monolithic electrodeposited nickel through the presence of nano-sized SiO_2 particles.

2. EXPERIMENTAL MATERIALS AND PROCEDURES

The galvanostatic deposition of pure Ni and co-deposition of nano-sized SiO_2 particles under direct current on copper foil were carried out by a recently developed technique [3,14,15]. After electroplating, the copper substrate was metallographic ground out to obtain freestanding samples for the subsequent mechanical and creep testing. The flat tensile specimens with a gauge length 25 mm and a width of 3.5 mm were machined from these freestanding

Corresponding author: V.Sklenicka, e-mail: sklen@ipm.cz

samples. The final thickness of the specimens was about 300 μm.

The tensile tests were carried out at room temperature, 373 and 473K and a strain rate of $1 \cdot 10^{-3}$ s^{-1} with a load cell resolution of 0.1 N. The constant stress tensile creep tests were carried out also at 293, 373 and 473K, respectively, and with the testing temperature continuously monitored and maintained constant to within ± 0.5K of the desired value. The applied stress ranged from 300 to 800 MPa. The creep tests were performed in purified argon in tensile creep testing machines, making it possible to keep the nominal stress constant to within 0.1% up to a true strain of about 0.35. The creep elongations were measured using a linear variable differential transducer. They were continuously recorded digitally and computer processed. Almost all the specimens were run to the final fracture.

Following mechanical testing, samples were prepared for examination by transmission electron microscopy (TEM) using a Philips CM 12 STEM microscope. Fractographic details were investigated by means of scanning electron microscopy using a Jeol SEM 6460 microscope.

3. RESULTS AND DISCUSSION

Fig. 1 shows TEM bright field and the corresponding SAD patterns for the as received electrodeposited pure nickel and its composite. TEM studies revealed mostly equiaxed grain shape. No voids and pores either in the grain interior or in the boundaries and no dislocations were observed in specimens of both materials. The grain size distribution of pure nickel was not unimodal and a number of grains were significantly larger than the average grain size of 300 nm (Fig. 1a). Growth twins were found to be prevalent in the microstructure. By contrast, very homogeneous microstructure was found for the particle-reinforced Ni-SiO_2 composite (Fig. 1b). An average grain size of 60 nm was estimated by quantitative image analysis. The nano-sized SiO_2 particles with the average particle size of 25 nm and 2 vol.% were homogeneously distributed in the nickel matrix. The pure nickel specimens from the room temperature tests did not show any grain growth. On the other hand, the post-creep specimens that had been tested at 373 and 473K show a slight increase in grain size observed by TEM.

The room temperature tensile tests showed a sharp increase in both the yield strength and ultimate tensile strength as compared with that of conventional-sized polycrystalline nickel. The yield strength increased from 103 MPa [16] in polycrystalline nickel to ~ 570 MPa in its electrodeposited counterparts as shown in Table 1. However, it should be stressed that even for the same material, the yield strength can be different upon changing load-

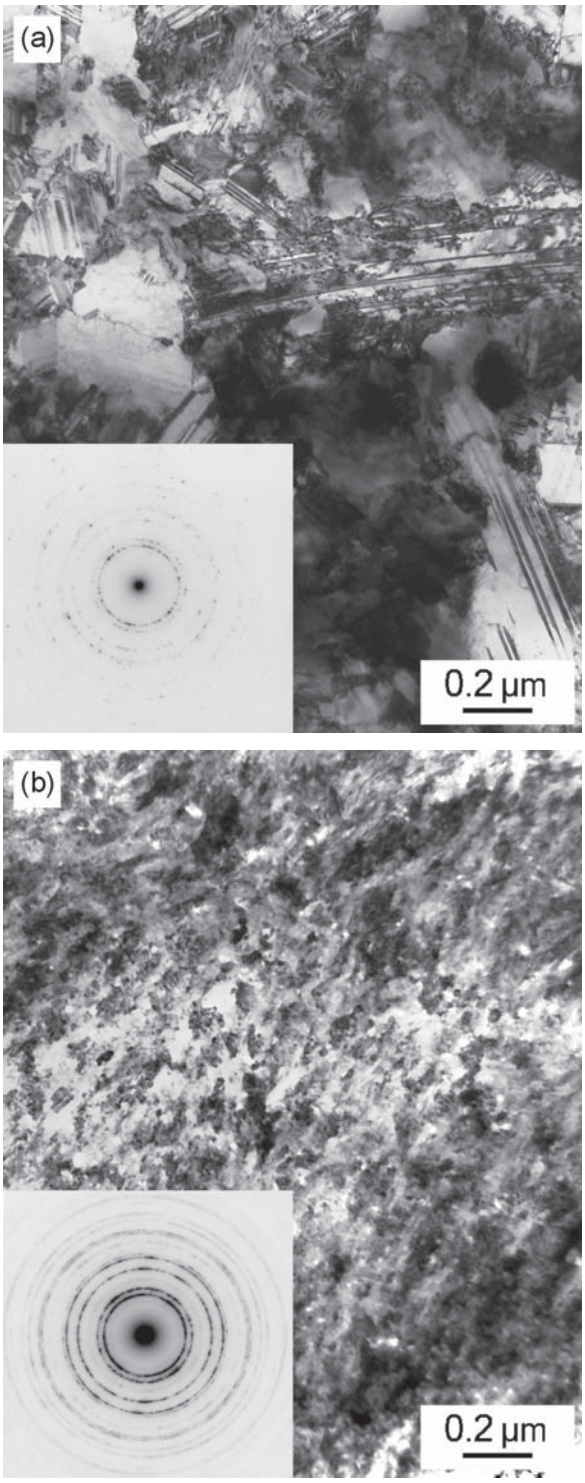

Fig. 1. TEM bright field micrographs and the corresponding SAD patterns for (a) the as-received electrodeposited pure nickel, and (b) its particle-strengthened composite.

Table 1. Summary of the tensile properties of the electrodeposits.

Deposits	Strain rate [s^{-1}]	Temperature [K]	E [GPa]	R_p 0.2% [MPa]	R_m [MPa]	A [%]
Ni-nc	10^{-3}	293	124	570	682	7.49
Ni-SiO_2	10^{-3}	293	142	568	857	5.30
Ni-nc	10^{-3}	373	129	456	592	11.51
Ni-SiO_2	10^{-3}	373	102	342	439	16.9
Ni-nc	10^{-3}	473	122	525	544	3.73
Ni-nc	10^{-3}	473	101	332	380	1.61
Ni-nc	10^{-3}	473	87	347	355	0.43
Ni-SiO_2	10^{-3}	473	92	309	380	8.4
Ni-SiO_2	10^{-3}	473	107	377	453	2.62
Ni-SiO_2	10^{-3}	473	93	360	430	2.01

ing rate [11,13], i.e. a lower loading rate gives rise to a lower yield strength. Also the determined values of the ultimate strength in the nickel electrodeposits are higher than those for conventional polycrystalline nickel (~ 400 MPa [16]). On the other hand, a significant reduction in tensile elongation A from that of polycrystalline nickel (A ~ 50%) to 5-7% as shown in Table 1. The testing temperature significantly influenced tensile behaviour; both the yield strength and ultimate tensile strength dropped with an increase in the testing temperature (Table 1).

Representative creep data are shown in Figs. 2a and 2b for the electrodeposited unreinforced pure nickel and its nanocomposite reinforced by nano-sized SiO_2 particles, respectively: all of these plots were obtained at room temperature, 373 and 473K over a range of values of the applied stress, σ, and they show (a) the variation of the minimum creep rate, $\dot{\varepsilon}_{min}$, with the stress and (b) the variation of the time to fracture, t_f , with the stress.

Several important conclusions may be reached from inspection of these data. First, it is important to note that the composite may exhibit better creep resistance than the unreinforced nickel. There is an order of magnitude difference in the minimum creep rate and the time to fracture between both the electrodeposits which is clearly demonstrated by the results at 373 and 473K (Fig. 2). However, the data for the composite at room temperature in Fig. 2 are insufficient to make a general conclusion being limited to an extremely narrow stress interval of the testing conditions as a result of very high stress sensitivity of the minimum creep rate and the creep lifetime. Second, as depicted from Fig. 2 the slopes and therefore the apparent stress exponents of the minimum creep rate $n = (\partial \ln\dot{\varepsilon}/\partial \ln\sigma)_T$ and the time to fracture $m = (\partial \ln t_f/\partial \ln\sigma)_T$ for both electrodeposits are the same. Third, the values of the exponents consistently decrease with increasing testing temperature.

The values of the strain to fracture in the unreinforced and reinforced electrodeposits seem to be essentially equal to each other and are typically ~10%. Representative SEM micrographs taken from the creep fracture surfaces of both electrodeposits are shown in Fig. 3. In both cases dimples were found on the fracture surface. However, the unreinforced nickel exhibits ductile fracture mode with rounded shallow surface feature (Fig. 3a), whereas some intergranular features with enhanced local ductility at grain boundaries regions can be seen in the composite (Fig. 3b).

The results available in the literature cannot be compared easily. The different techniques used to produce the materials result in widely different internal structures and processing – induced artefacts, such as contamination, porosity and residual stress. These differences render it difficult to identify the mechanisms responsible for differences in tensile and creep properties. Another common problem is that only a limited amount of material is available to perform testing. Further, mechanical and especially creep testing involve different specimen design, loading methods and elongation monitoring techniques.

Several earlier reports describe some of the creep data obtained with nickel processed by elec-

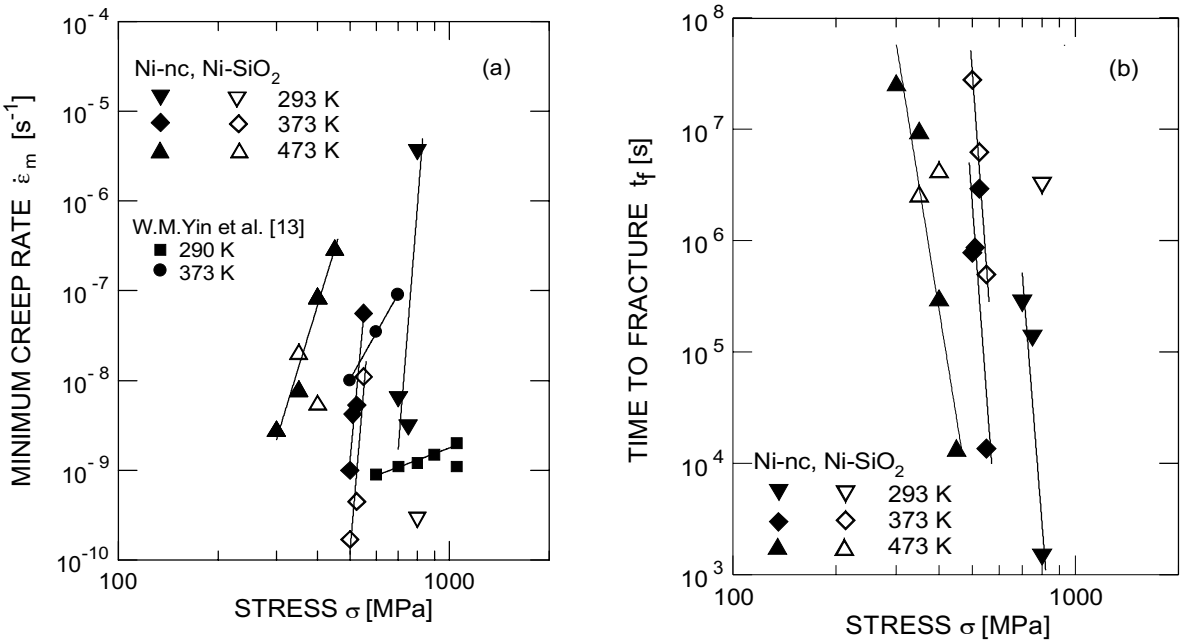

Fig. 2. Stress dependences of (a) minimum creep rates, and (b) times to fracture for electrodeposited pure nickel (Ni-nc) and its composite. The experimental data for pure electrodeposited nickel with grain size of 30 nm reported by Yin *et al*. [13] are plotted in Fig. 2a.

trodeposition. The uniaxial tensile and creep behaviour of pure nanocrystalline nickel with grain size 6 - 40 nm prepared by pulse electrodeposition technique at room temperature were studied by Wang *et al.* [5]. The results of this study indicates that the room temperature creep by nanocrystalline nickel electrodeposits is controlled by the grain boundary sliding mechanism and the contribution to the total creep strain due to diffusion along the intercrystalline components becomes significant only at smaller grain sizes (< 20 nm). Experimentally determined value of the stress exponent of the creep rate n was of ~ 1.18 for the samples with grain 6 nm in size. The experimental data for 40 nm indicate a transition between deformation mechanisms. Wang *et al.* [5] suggested that power law or dislocation creep ($n \geq 5$) becomes the predominant deformation mechanism at high stresses which explains the value of $n \sim 5.9$ determined experimentally.

Yin *et al.* [13] studied the creep behaviour of electrodeposited nanocrystalline nickel with grain size of about 30 nm at room temperature and 373K in a stress interval 500 – 1050 MPa. The experimental results showed that significant creep deformation occurred at room temperature at an initial applied stress of 600 MPa or higher. The stress exponent in Fig. 2a is ~1.1 for room temperature creep while

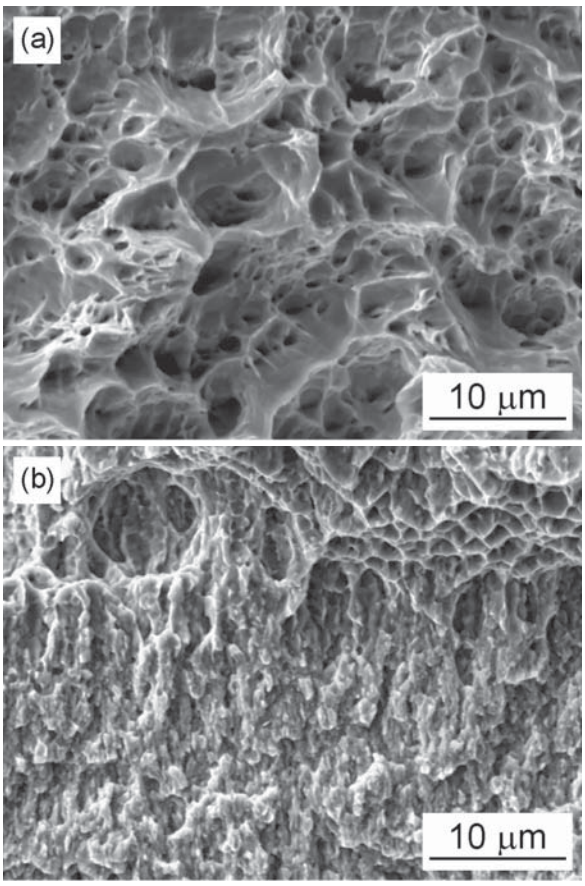

Fig. 3. SEM micrographs of creep fracture surfaces of (a) electrodeposited pure nickel, and (b) its composite (creep at 473K and 400 MPa).

it is 6.5 for creep at 373K. These results indicate that the creep mechanisms at room temperature may be governed by diffusional creep (most probably by a Coble creep mechanisms [17]), in which enhanced diffusion along intercrystalline components including grain boundaries, triple junctions and quadrupole nodes control the mass transport process. However, the creep deformation mechanisms might change from a diffusion-controlled process at room temperature to a more complex mechanism above ambient temperature as indicated the value of 6.5 for 373K.

In this work the minimum creep rate is related to the applied stress through a power-law in which the values of the stress exponent n are ~ 46.7, 39.1 and 12 for temperature 293, 373, and 473K, respectively (Fig. 2a). Such high values are inherent to creep behaviour in precipitation and dispersion strengthened alloys [17], however, very similar values were recently found for pure aluminium at low temperature creep experiments [18], though a dominant mechanism has not yet been identified.

The improved creep resistance of the composite can be explained by the fact that the matrix sheds load to the reinforcement during creep and by the interaction with dislocation motion. However, the very close values of the stress exponent of the minimum creep rate in the monolithic nickel and the composite indicate that creep deformation in the composite is controlled by flow in the matrix material.

4. CONCLUSIONS

The tensile properties and the creep behaviour of the electrodeposited pure ultrafine-grained nickel and its nanocomposite reinforced by 2 vol.% nano-sized SiO_2 particles were studied at temperatures 293, 373 and 473K. It was found that tensile properties of composite were not considerably improved by reinforcement in contrast to its creep resistance. Better understanding of the deformation processes especially grain boundary-related processes at ultrafine-grained and nanoscale grain sizes at ambient and elevated temperatures is strongly needed.

ACKNOWLEDGEMENTS

This work was supported by the Ministry of Education of the Czech Republic under the Czech-German Programme KONTAKT ME 661 and by the Academy of Sciences of the Czech Republic under the Institutional Research Plan AV0Z20410507.

REFERENCES

[1] C.Suryanarayana and C.C.Koch, In: *Non-equilibrium Processing of Materials*, ed. by C.Suryanarayana (Elsevier Science Ltd., Oxford, 1999) p. 317.
[2] K.T.Aust, G.H.Mibbard, G.Palumbo and U.Erb // *Z.Metallkd*. **94** (2003) 1066.
[3] B.Muller and H.Ferkel // *Nanostructured Mater*. **10** (1998) 1285.
[4] E.Arzt // *Acta Mater.* **46** (1998) 5611.
[5] N.Wang, Z.Wang, K.T.Aust and U.Erb // *Acta Mater.* **45** (1997) 1655.
[6] U.Klement, U.Erb, A.M. El-Sherik and K.T.Aust // *Mater.Sci.Eng. A* **203** (1995) 177.
[7] Y.M.Wang, S.Cheng, Q.M.Wei, E.Ma, T.G.Nieh and A.Hamza // *Scripta Mater.* **51** (2004) 1023.
[8] N.Wang, Z.Wang, K.T.Aust and U.Erb // *Mater.Sci.Eng. A* **237** (1997) 150.
[9] L.Lu, S.X.Li and K.Lu // *Scripta Mater.* **45** (2001) 1163.
[10] F.D.Torre, H.Van Swygenhoven and M.Victoria // *Acta Mater.* **50** (2002) 3957.
[11] R.Schwaiger, B.Moser, M.Dao, N.Chollacoop and S.Suresh // *Acta Mater.* **51** (2003) 5159.
[12] F.Ebrahimi, G.R.Bourne, M.S.Kelly and T.E.Matthews // *Nanostructured Mat.***11** (1999) 343.
[13] W.M.Yin, S.H.Whang, R.Mirshams and C.H.Xiao // *Mater.Sci.Eng. A* **301** (2001) 18.
[14] H.Ferkel, B.Muller and W.Riehemann // *Mater.Sci.Eng. A* **234-236** (1997) 474.
[15] J.F.Castagnet, *PhD. Thesis* (TU Clausthal, 2005).
[16] *ASM Metals Handbook* (ASM International, Metals Park, OH, 2,1993).
[17] J.Cadek, *Creep in Metallic Materials* (Elsevier Science Publishers, Amsterdam, 1988).
[18] P.J.Krajewski, In: *Deformation, Processing and Properties of Structural Materials*, ed by. E.M.Taleff *et al.* (TMS, Warrendale, 2000) 207.