Superplasticity and Residual Tensile Properties of a Microduplex Copper-Nickel-Zinc Alloy

R. D. SCHELLENG AND G. H. REYNOLDS

Structural superplasticity in two phase alloys of the copper-nickel-zinc system (nominal composition in wt. pct Cu-15 Ni-38 Zn-0.2 Mn) occurs over a wide range of strain rates in the temperature range 850 to 1050°F (454 to 565°C). The upper temperature limit for superplastic behavior in this system is determined by the reversion of the fine-grained two-phase structure to a single phase structure in which extensive grain growth is possible. Residual room temperature tensile properties and microstructure of the microduplex alloy after superplastic straining have been studied as a function of test temperature and total superplastic strain. At test temperatures sufficiently removed from the phase transformation temperature, the high tensile properties and fine microstructure of the starting material are essentially retained after superplastic strains approaching 200 pct. In the immediate vicinity of the phase transformation temperature, rapid degradation of the microduplex structure occurs during superplastic deformation with a consequent severe degradation of the residual room temperature tensile properties.

THE phenomenon of structural superplasticity has been observed in a variety of fine grained alloy systems and has been the subject of two recent review articles.[1,2] It has been suggested by several authors[1-3] that the phenomenon may be commercially useful in low pressure forming of complex shapes. The initial work on superplasticity in copper base alloys has centered on the aluminum bronzes[5-8] and binary brasses.[7,9] The present work reports on the occurrence of structural superplasticity in a two phase alloy of the copper-nickel-zinc system (nominal composition in wt pct: copper-15 Ni-38 Zn-0.2 Mn). This alloy offers the combined benefits of low superplastic forming temperatures and exceptionally high mechanical properties in the annealed (recrystallized) two-phase alloy.[4]

Cavitation and/or grain growth sometimes occur during superplastic extension of a variety of alloys and various authors have investigated the problem.[2,8,9,10] Davis *et al.*[1] have acknowledged the need for characterization of the properties of superplastic alloys after superplastic deformation since extensive cavitation would result in poor properties. The present work includes an examination of the microstructural changes accompanying superplastic extension as influenced by test temperature, total strain, and alloy composition. In addition, the room temperature (residual) tensile properties of the alloy after superplastic extension have been determined and related to the residual microstructure.

EXPERIMENTAL PROCEDURES

Processing steps to produce the microduplex alloys are similar to those previously reported[4] and are listed in Table I. The microduplex structure is a fine grained intermixture of the fcc α and bcc β phases having an interphase spacing of approximately 1 μm. The structure is produced by simultaneous recrystallization and β phase precipitation from a heavily cold worked metastable α structure. A replica electron micrograph of the lightly etched microduplex structure is shown in Fig. 1. Compositions of the experimental heats prepared for the present experiments are shown in Table II. Two different deoxidants were explored since this factor had been found to be of importance in another alloy.[9]

Table I. Preparation Sequence for Experimental Microduplex Copper-Nickel-Zinc Alloys

1. Air induction melt virgin raw materials.
2. Deoxidize with Mg or Ti preceded by Mn.
3. Cast as 30 lb. (13.6 Kg), 4 X 4 in. (10 X 10 cm) ingots.
4. Forge and hot roll at 1500°F(815°C) to 0.5 in. (1.3 cm) plate.
5. Solution anneal at 1300°F(705°C) and water quench to retain single phase fcc alpha structure.
6. Cold roll metastable alpha 85 pct.
7. Microduplex anneal at 950°F(510°C) to recrystallize alpha and precipitate beta.

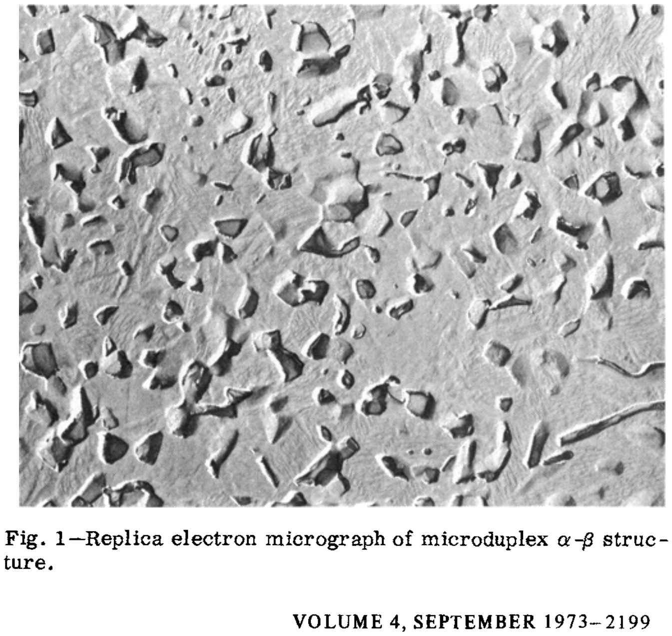

Fig. 1—Replica electron micrograph of microduplex α-β structure.

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Strain rate sensitivity tests[10] were performed on strip tensile specimens [0.25 × 1.125 in. (0.63 × 2.86 cm) gage section] in an argon atmosphere at temperatures ranging from 850 to 1200°F (454 to 649°C) on an Instron testing machine. The steady state loads at various constant crosshead speeds were taken from a continuous record of load vs time. True flow stresses and true strain rates were computed from these data.

Additional strip specimens were superplastically extended to total (engineering) strains of 20 to 316 pct with the "Gleeble"*,[11] an apparatus which subjects specimens to a programmed time-temperature-strain cycle. By trial and error, it was found that a 1 × 9 in. (2.5 × 21.6 cm) strip specimen (heated electrically to the test temperature) provided reasonably uniform deformation over a 1 in. (2.5 cm) section at the center of the strip. Scribe marks were placed in this section of the strip before the start of each test so that total strain and average strain rate (total strain divided by test duration) in the section were known. Specimens were extended at 875, 975, and 1075°F (469, 524, and 580°C). Strain rates in the 1 in. section varied somewhat from test to test but the average strain rate was approximately 0.14 per min.

Room temperature tensile test specimens were cut from the superplastically extended strips with their 0.25 × 1.125-in. (0.63 × 2.86 cm) gage sections located in the hot-deformation zone. The surfaces of the tensile specimen were ground flat and parallel to eliminate slight surface roughening occurring during superplastic extension. These tensile specimens were tested at room temperature to determine residual mechanical properties. After testing, the gage section was examined metallographically for evidence of cavitation and grain growth.

EXPERIMENTAL RESULTS AND DISCUSSION

Superplasticity

Between 850 and 1050°F (454 and 565°C) classical superplastic behavior is observed as evidenced by the high strain rate sensitivity exponents (*m* values) shown by the true flow-stress/true strain-rate data plotted in Fig. 2. At 1100°F (594°C), the flow-stress/strain-rate curve is somewhat erratic and the strain rate sensitivity exponent has decreased in value. This temperature corresponds to the $\alpha + \beta \rightarrow \alpha$ solvus for this composition as determined by Schramm.[12] Above this temperature, the microduplex structure reverts to a single phase α structure in which extensive grain growth is possible due to the absence of a second phase. Such behavior is reflected in the 1150°F (622°C) data. The flow-stresses are higher, particularly at low strain rates, and the strain rate sensitivity exponent has decreased to a value where superplastic behavior would be unlikely. Experimental data obtained at 1200°F (649°C) (not included in the figure for the sake of clarity) parallel those obtained at 1150°F (622°C), but at lower flow stresses.

Fig. 3 shows the true flow stress data of Fig. 2 replotted as a function of test temperature and true strain rate. At the lowest strain rate (0.002 per min), the flow stress decreases steadily with increasing temperature in the superplastic temperature range reaching a minimum value of 500 psi (3.44 MN per sq m) at 1100°F (594°C). The flow stress then increases sharply as the phase transformation temperature is exceeded. Growth of the α grains, permitted by the solution of the β phase, increases the flow stress. Examination of the two curves for higher strain rates shows that the phase transformation effect is less pronounced and suggests that the actual phase transformation temperature may vary slightly with stress or strain rate, particularly

Table II. Compositions of Experimental Copper-Nickel-Zinc Alloys (Wt. Pct)

Heat No.	Cu	Ni	Zn	Mn	Ti	Mg
1	Bal.	14.0	38.8	0.10	0.03	–
2	Bal.	14.6	37.2	0.17	0.04	–
3	Bal.	15.1	38.0	0.12	–	0.02

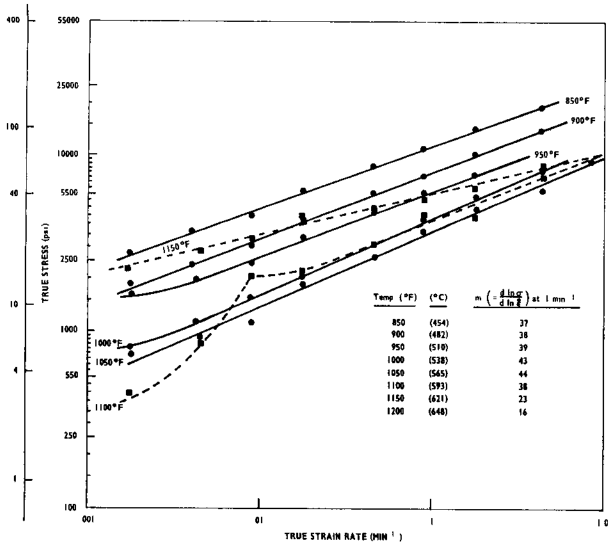

Fig. 2—Flow stress vs strain rate for microduplex Cu-Ni-Zn alloy in the superplastic and nonsuperplastic temperature region.

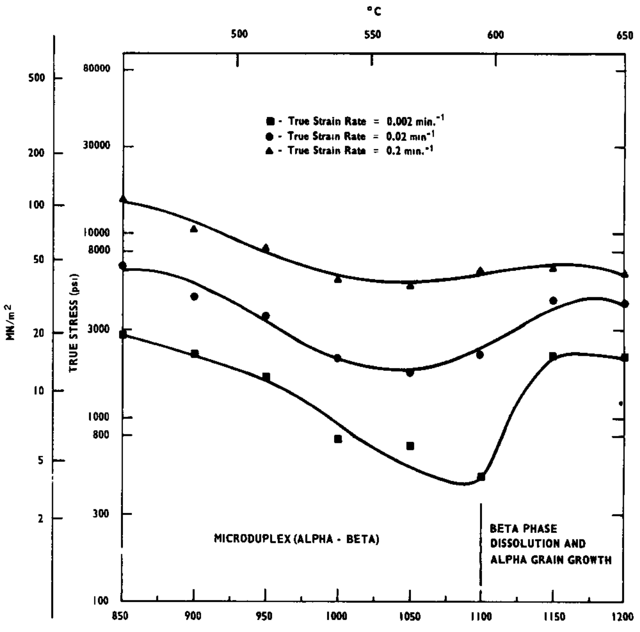

Fig. 3—Flow stress vs test temperature for microduplex Cu-Ni-Zn alloy in the superplastic and nonsuperplastic temperature region.

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Table III. Residual Room Temperature Tensile Properties of Microduplex Cu-Ni-Zn Alloy* After Superplastic Extension

Heat No.	Deoxidant	Test Temp. °F (°C)	Average Strain Rate at Temperature (min^{-1})	Total Strain at Temp. (pct)	0.2 pct YS ksi (MN per sq. m)	UTS, ksi (MN per sq m)	Pct Elong.
2	Ti	875(469)	0.066	20	88.7(612)	110.5(763)	22
2	Ti	875(469)	0.110	43	88.8(613)	111.2(768)	22
2	Ti	875(469)	0.127	76	86.9(599)	109.3(755)	21
2	Ti	875(469)	0.131	92	86.9(599)	108.7(750)	19
2	Ti	875(469)	0.179	143	85.8(591)	105.3(727)	16
2	Ti	875(469)	0.316	316	80.0(552)	85.4(589)	2
3	Mg	875(469)	0.094	28	85.8(591)	110.8(764)	24
3	Mg	875(469)	0.089	53	87.3(602)	111.8(770)	21
3	Mg	875(469)	0.179	143	87.2(601)	103.9(716)	12
3	Mg	875(469)	0.204	143	103.9(715)	127.5(880)	14
2	Ti	975(524)	0.110	33	85.1(588)	106.1(732)	24
2	Ti	975(524)	0.108	52	83.8(578)	103.8(715)	21
2	Ti	975(524)	0.130	52	85.5(590)	104.5(721)	24
2	Ti	975(524)	0.135	74	82.6(570)	102.0(704)	22
2	Ti	975(524)	0.200	120	83.0(573)	102.2(706)	21
2	Ti	975(524)	0.247	173	79.8(551)	88.7(612)	9
3	Mg	975(524)	0.140	28	83.7(578)	107.7(743)	24
3	Mg	975(524)	0.143	43	81.2(560)	105.2(726)	24
3	Mg	975(524)	0.118	59	82.3(568)	105.0(724)	22
3	Mg	975(524)	0.154	108	80.1(553)	98.9(682)	15
3	Mg	975(524)	0.195	156	76.4(527)	95.0(655)	16
3	Mg	975(524)	0.255	204	76.2(525)	80.6(556)	3
1	Ti	1075(580)	0.070	21	17.9(124)	24.1(166)	31
1	Ti	1075(580)	0.095	66	16.1(111)	22.8(157)	32
1	Ti	1075(580)	0.084	126	11.5(79.4)	13.3(92)	3
1	Ti	1075(580)	0.102	156	12.2(84.2)	13.7(95)	2
3	Mg	1075(580)	0.094	28	72.6(501)	96.4(665)	28
3	Mg	1075(580)	0.088	53	68.8(474)	92.0(635)	30
3	Mg	1075(580)	0.115	92	68.9(475)	90.9(627)	28
3	Mg	1075(580)	0.100	100	62.3(430)	85.6(591)	25

*Starting properties of annealed microduplex alloy 84 ksi (580 MN per sq m) YS, 102 ksi (704 MN per sq. m) UTS, 25 pct elongation.

in the strain rate range 0.002 to 0.02 per min.

Activation energies for superplastic deformation of 34.2 and 35.0 kcal per mole were determined from Arrhenius plots of strain rate vs inverse temperature for the temperature range 850 to 1050°F (454 to 565°C) (0.6 to $0.7T_m$) at true stress levels of 3000 and 6000 psi (20.7 and 41.4 MN per sq m) respectively. Volume diffusion activation energies for fcc copper-base alloys containing approximately 18 to 21 wt pct Ni and 24 to 31 pct zinc for all three species are in the range 43 to 47 kcal per mole.[13] Thus, the experimentally determined activation energy for superplastic deformation does not correspond to volume diffusion of any constituent atom in the fcc α phase. As Hayden *et al.*[14] have shown, the activation energy for superplasticity may vary with the temperature regime of the experiments (*i.e.*, with the mechanism) and the low values obtained in the present work may correspond to diffusion via a short circuiting path or perhaps to rate-controlling creep processes in the bcc β phase. There are not sufficient experimental data yet available on diffusion in the ternary to relate the experimental activation energy to a particular process or to say in which phase the deformation processes are rate controlling.

Residual Tensile Properties

After superplastic extension at 875 or 975°F (469 or 524°C) to strains approaching 200 pct the properties of

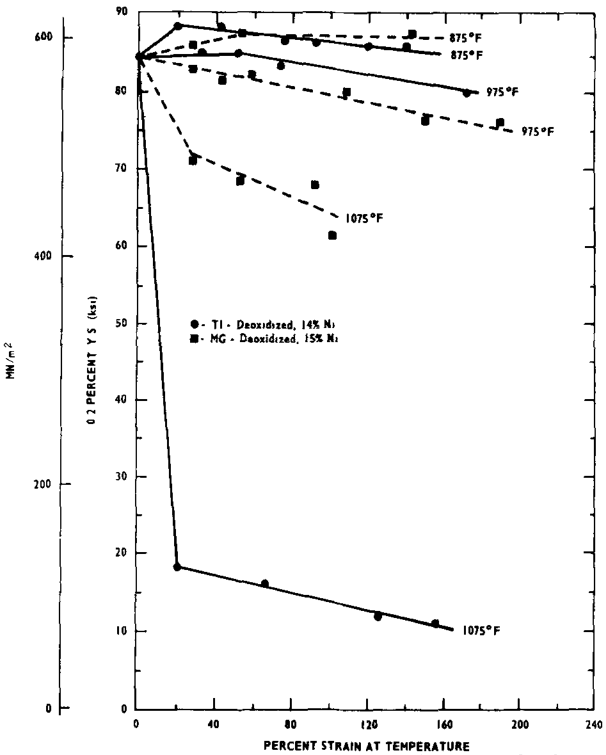
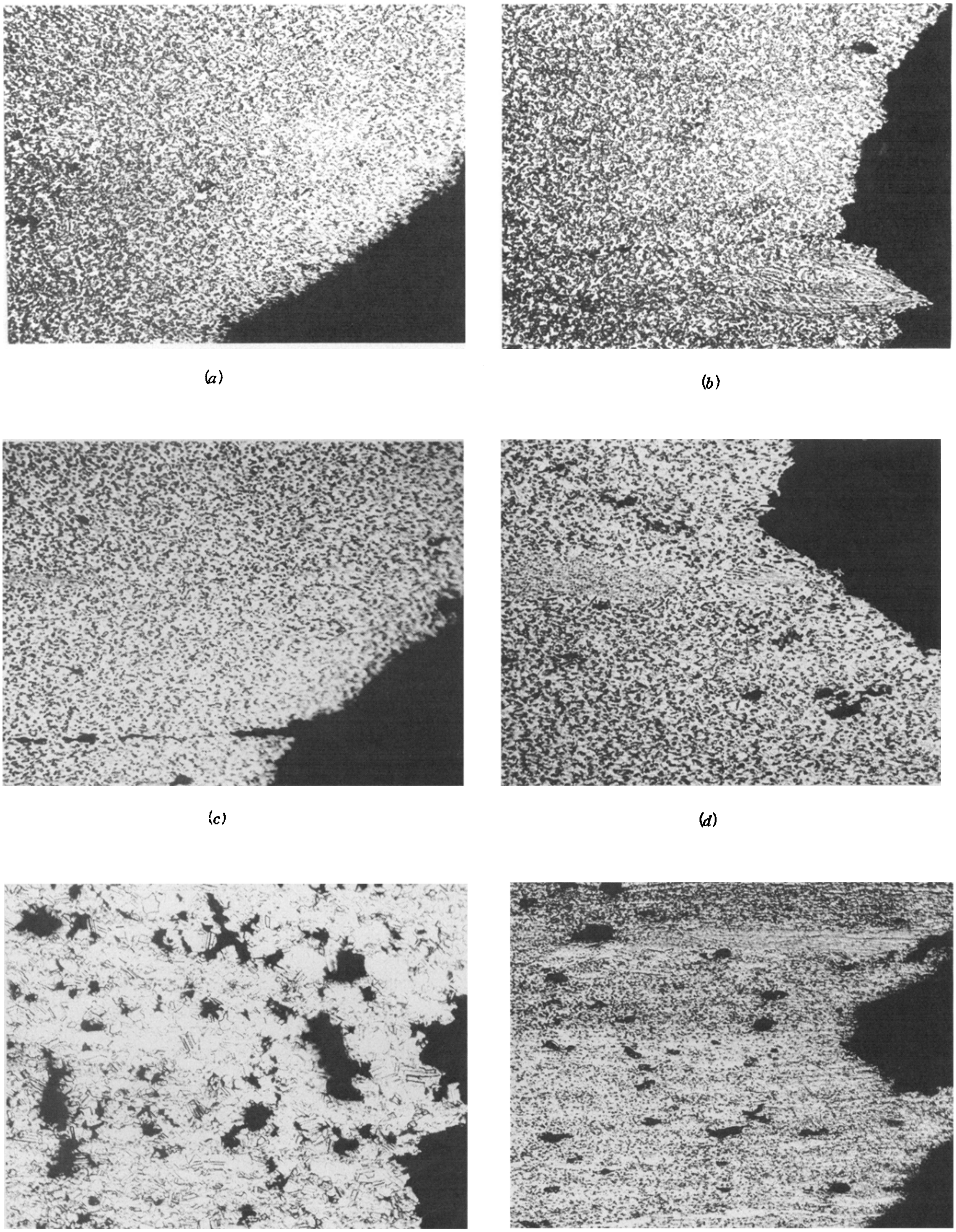


Fig. 4—Room temperature (residual) yield strength of microduplex Cu-Ni-Zn alloy after superplastic extension at 875, 975, and 1075°F (469, 524, and 580°C).



(a)

(b)

(c)

(d)

(e)

(f)

Fig. 5—Microstructures after indicated superplastic strain and subsequent tensile testing at room temperature. Tensile axis is horizontal; (*a*) 143 pct, 875°F (469°C), Heat No. 2 (*b*) 143 pct, 875°F (469°C), Heat No. 3; (*c*) 173 pct, 975°F (524°C), Heat No. 2; (*d*) 156 pct, 975°F (524°C), Heat No. 3; (*e*) 156 pct, 1075°F (580°C), Heat No. 2; (*f*) 92 pct, 1075°F (580°C), Heat No. 3.

the material are equivalent to those of the starting material as shown in Table III and Fig. 4. These temperatures are well within the α-β phase field and the microduplex structure is retained without β phase dissolution or excessive growth of the α phase grains as shown in Figs. 5(*a*) to (*d*). Only a small volume fraction of voids is generated in the microstructure by these superplastic strains. Larger strains generate a larger volume fraction of voids which decrease the residual tensile properties, particularly tensile elongation as seen in Table III. The voids presumably act as severe internal stress concentrators due to their irregular shape. There appears to be no particular effect of the specific deoxidants, Mg or Ti, on residual tensile properties or the severity of cavitation in the microstructure.

After extension at 1075°F (580°C), reductions in the residual strength of both alloys occur after strains of 20 to 30 pct. Residual microstructures of these samples are shown in Figs. 5(*e*) and (*f*). The test temperature, 1075°F (580°C), corresponds closely to the phase transformation temperature and slight variations in alloy composition or test temperature could affect the structural stability of the alloy at this temperature. Attempts to superplastically form complex shapes using the microduplex alloy should avoid the critical temperature range close to the phase transformation temperature. Even though forming stresses are somewhat higher in the lower forming temperature ranges, structural stability of the alloy and excellent residual tensile properties are achieved.

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