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Effect of Heat Treatment on the Specific Heat Capacity of Nickel-Based Alloys[1]

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Alloy-718 and Udimet alloy 720 are gamma prime strengthened superalloys with excellent mechanical and thermal properties at elevated temperatures, as well as at cryogenic temperatures. The nickel-based alloys were improved to be resistant to creep and become stronger by changing the heat-treatment conditions. The measurement of the specific heat capacity of a nickel-based alloy is a very useful tool to investigate the effect of heat treatment. The specific heat capacity of nickel-based alloys Alloy-718 and Udimet alloy 720 were measured using a differential scanning calorimeter in the temperature range of 100 – 1000 K. The specific heat capacity of the nickel-based alloys increases monotonically with temperature; however, above 800 K, it is strongly dependent on the heat treatment conditions and it is thought to be influenced by the precipitation phase (γ', γ''). Optical and scanning electron microscopies are used to investigate the microstructure of the phases. The microstructures of the precipitates are examined.

KEY WORDS: Alloy-718; differential scanning calorimeter (DSC); heat treatment; nickel-based alloy; precipitation; Udimet alloy 720.

1. INTRODUCTION

A variety of nickel-based alloys are currently used for gas turbine components, aircraft engines, nuclear vessel components, cryogenic storage tanks,

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etc. [1–5]. Among them, Alloy-718 and Udimet alloy 720 (U-720) are gamma prime strengthened superalloys with excellent mechanical and thermal properties at elevated temperatures as well as at cryogenic temperatures. Alloy-718 is a precipitation hardened nickel-based alloy designed to display exceptionally high yield, tensile, and creep-rupture properties at temperatures up to 977 K. The age-hardening response of Alloy-718 permits annealing and welding without spontaneous hardening during heating and cooling processes. Alloy-718 has excellent weldability as compared with the nickel-based superalloys hardened by aluminum and titanium. Alloy-718 is used in aircraft engine parts and high-speed airframe parts such as wheels, buckets, spacers, and high temperature bolts and fasteners [6]. U-720 is a solid solution strengthened with tungsten and molybdenum and precipitation hardened with titanium and aluminum. The alloy combines high strength with metallurgical stability as demonstrated by excellent impact strength retention after long exposures at elevated temperatures, and the alloy is useful in gas turbine blade and disc applications [7].

The nickel-based alloys were improved to be resistant to creep and becomes stronger by changing the heat treatment conditions. The measurement of the specific heat capacity of a nickel-based alloy is a useful tool to investigate the effect of heat treatment. In this study, the specific heat capacities of Alloy-718 and U-720 have been measured by a DSC (differential scanning calorimeter) in the temperature range of 100 – 1000 K. The samples were heat treated for a precipitation phase conditioned solution and/or precipitation treatment, and these heat treatment conditions are commonly used in these alloys.

2. EXPERIMENTAL PROCEDURES

2.1. Sample Preparation

Alloy-718 and U-720 used in this work, products of Teledyne Allvac, were obtained in the form of billets. Alloy-718 and U-720 were nickel-based superalloys. The chemical compositions of these specimens are shown in Table I. These superalloys are composed of nickel, chrome, and iron or cobalt.

Table I. Chemical Compositions (in Mass%) of the Superalloys

Sample	Ni	Cr	Co	Fe	Mo	W	Al	Ti	Si
Alloy-718	Balance	18.6	0.3	17.7	7.0	0.6	0.9	1.1	6.3
Udimet-720	Balance	15.8	14.7	0.2	3.0	1.0	2.8	5.2	0.3

The superalloys were heat treated with a standard several step process prior to measurement. The samples were solution treated at heat treatment conditions such as ST (solution treatment) and PT (precipitation treatment). The superalloys were precipitation-hardened nickel-based alloys designed to display exceptionally high yield strengths. The Alloy-718 samples were placed inside a ceramic boat and solution treated at 1253 K for 4 h followed by water quenching (ST). The samples were then held at 993 K for 8 h followed by furnace cooling (PT). While the U-720 specimens were solution treated at 1378 K for 4 h and water cooled to room temperature. The PT-1 involved annealing at 1033 K for 8 h followed by air cooling to room temperature, and the annealed heat treatment was at 923 K for 24 h and then air cooled. The PT-2 precipitation treatment involved solution treatment followed by annealing at 923 K for 24 h followed by air cooling to room temperature, and the annealed heat treatment was at 1033 K for 8 h and air cooled. Various heat treatments are listed in Table II.

The microstructure of the superalloys was observed with an optical microscope and a scanning electron microscope (SEM). In order to observe the changes of the morphology and precipitation, the samples were etched with Kalling's reagent (5 $CuCl_2$ + 100 ml HCl + 100 ml ethanol) for Alloy-718 and U-720 [8].

2.2. Specific-Heat-Capacity Measurements [9]

The specific heat capacity (C_p) was measured with a DSC (Perkin–Elmer, Pyris 1) over a temperature range from 100 to 1000 K. The measurements were carried out with a heating rate of $5\,K \cdot min^{-1}$ in a helium atmosphere at low temperatures, 100–300 K, and in a nitrogen atmosphere

Table II. Heat Treatments of the Samples

Specimens	Heat treatment conditions
Alloy-718	AR: as received, no heat treatment ST:1253 K/4 h → water-cooled PT:ST → 993 K/8 h → furnace-cooled → 893 K/8 h → air-cooled
Udimet-720	AR: as received, no heat treatment PT-1:1378 K/4 h → water-cooled → 1033 K/8 h → air-cooled → 923 K/24 h → air-cooled PT-2:1378 K/4 h → water-cooled → 923 K/24 h → air-cooled → 1033 K/8 h → air-cooled

at high temperatures, 300–1000 K, with a flow rate of 30 ml · min^{-1}. The NIST synthetic sapphire, SRM 720, was used as a reference material. The standard deviation in measurements of the specific heat capacity is estimated to be 2%. The specimens were cut into disks 5 mm in diameter and about 2 mm in thickness.

3. RESULTS AND DISCUSSION

The microstructures of Alloy-718 and U-720 are shown in the micrographs of Figs. 1 and 2. The basic microstructure consists of the γ matrix, large γ' precipitate, and a dispersion of a fine secondary γ' precipitate. The superalloy precipitated γ' and γ'' phases were produced by typical heat treatment consisting of solution annealing and aging. The nickel-based alloys were strengthened by these gamma prime (γ') and gamma double prime (γ'') precipitate phases. Gamma prime, the first of the two phases to precipitate during aging, is a coherent, ordered Ni_3 (Al, Ti, Nb) phase and has a spherical or cuboidal morphology. Gamma double prime, which nucleates and coarsens on the γ' particles, is a coherent but misfitting and ordered metastable Ni_3 (Al, Ti, Nb) phase and appears as disc-shaped particles [8].

The measured specific heat capacities of Alloy-718 and U-720 are shown in Figs. 3(a) and (b). The specific heat capacities of Alloy-718 and U-720 were measured at steps of 20 K. We measured three times for each sample and the reproducibility and uncertainty were within 2%. The specific heat capacity of the nickel-based alloys increases monotonically with temperature, but it was strongly dependent on heat treatment conditions above 800 K. It was thought to be influenced by the precipitation phase (γ', γ''). Alloy-718 and U-720 showed anomalous specific heat capacities around 800 K, this temperature may represent the γ', γ'' precipitation phases. The nickel-based superalloys were precipitation hardened, especially at temperatures between 800 and 1100 K, indicating the formation of the γ' and/or γ'' phases [1]. Alloy-718 and U-720 show the same trends, but the specific heat capacity of Alloy-718 is higher than for U-720 above 900 K.

The measured specific heat capacities of Alloy-718 and U-720 from 800 to 1000 K are shown in Figs. 4(a) and (b). The specific heat capacity of Alloy-718 shows no difference for AR and ST heat treatments, but the PT specimen gives values 2–10% lower than those for AR since the PT condition had more precipitation phases. The specific heat capacity of U-720 at high temperatures decreases by only 2% with heat treatment and longer annealing time.

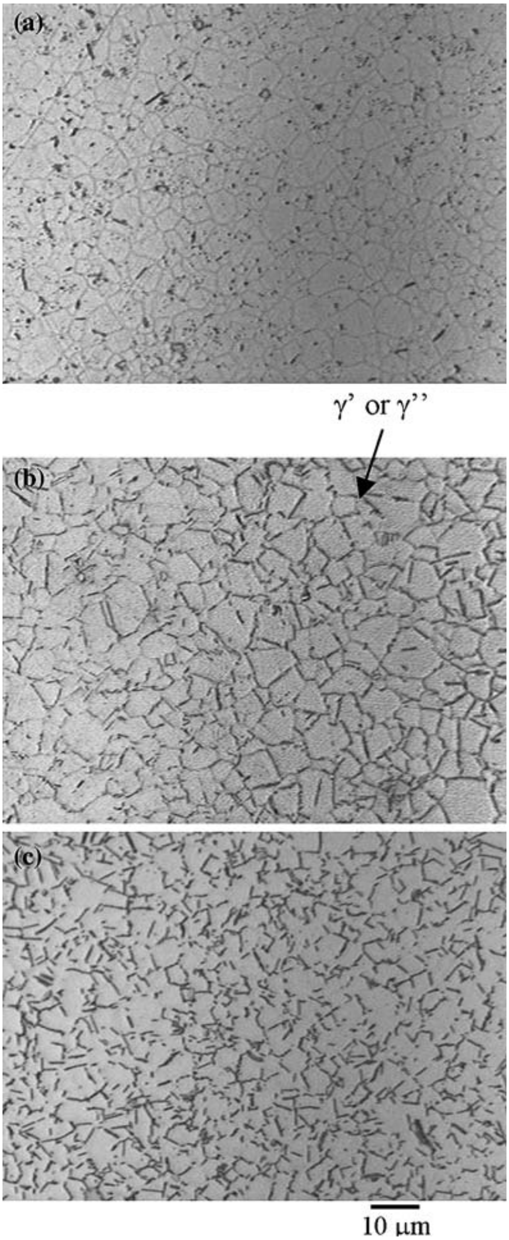

Fig. 1. Micrographs illustrating the structure of Alloy-718 with different heat treatment conditions; (a) Alloy-718 (AR), (b) Alloy-718 (ST), and (c) Alloy-718 (PT).

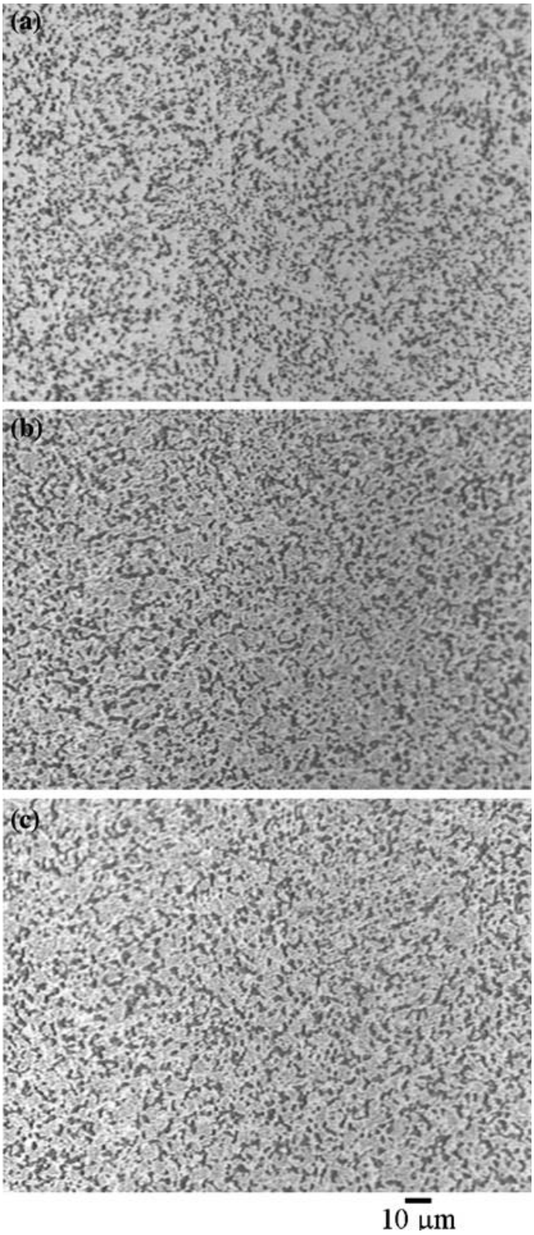

Fig. 2. Micrographs illustrating the structure of Udimet-718 with different heat treatment conditions; (a) U-720 (AR), (b) U-720 (PT-1), and (c) U-720 (PT-2).

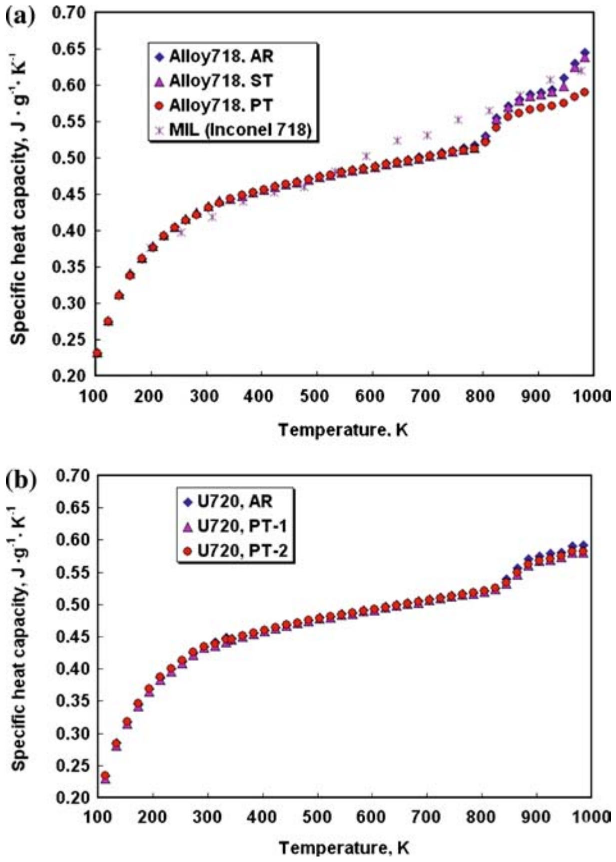

Fig. 3. Specific heat capacity of (a) Alloy-718 and (b) Udimet alloy 720.

These results show small deviations, but within 5%, when compared to the MIL data for Inconel 718 [10]. The measured specific heat capacities of Alloy-718 and U-720 are shown in Tables III and IV. From polynomial least-squares fits we obtain

Alloy-718:

$$C_p(\mathrm{J \cdot g^{-1} \cdot K^{-1}}) = -0.20761 + 6.7421 \times 10^{-3} T - 3.1271 \times 10^{-5} T^2 \\ + 8.1231 \times 10^{-8} T^3 - 1.1971 \times 10^{-10} T^4 \\ + 9.3663 \times 10^{-14} T^5 - 3.0156 \times 10^{-17} T^6 \quad 100\,\mathrm{K} < T < 800\,\mathrm{K};$$

Table III. Specific Heat Capacity of Alloy-718.

Temperature (K)	Alloy 718-AR ($J \cdot g^{-1} \cdot K^{-1}$)	Alloy 718-ST ($J \cdot g^{-1} \cdot K^{-1}$)	Alloy 718-PT ($J \cdot g^{-1} \cdot K^{-1}$)
103.4	0.2316	0.2326	0.2307
123.4	0.2754	0.2763	0.2745
143.4	0.3114	0.3126	0.3101
163.3	0.3395	0.3409	0.3381
183.3	0.3615	0.3624	0.3606
203.5	0.3778	0.3788	0.3768
223.4	0.3931	0.3941	0.3921
243.4	0.4045	0.4054	0.4037
263.5	0.4152	0.4163	0.4141
283.5	0.4229	0.4244	0.4213
303.5	0.4321	0.4333	0.4309
323.5	0.4389	0.4408	0.4370
344.2	0.4434	0.4435	0.4434
364.3	0.4483	0.4480	0.4483
384.2	0.4523	0.4519	0.4522
404.2	0.4560	0.4557	0.4560
424.4	0.4607	0.4599	0.4605
444.4	0.4642	0.4643	0.4640
464.3	0.4671	0.4668	0.4668
484.5	0.4706	0.4704	0.4704
504.5	0.4746	0.4738	0.4738
524.5	0.4767	0.4758	0.4760
544.6	0.4809	0.4799	0.4798
564.5	0.4833	0.4823	0.4822
584.5	0.4865	0.4853	0.4853
604.6	0.4888	0.4874	0.4871
624.6	0.4927	0.4915	0.4914
644.7	0.4950	0.4937	0.4935
664.7	0.4978	0.4968	0.4966
684.7	0.5007	0.4990	0.4987
704.7	0.5043	0.5028	0.5023
724.7	0.5070	0.5054	0.5051
744.9	0.5102	0.5091	0.5082
764.7	0.5134	0.5112	0.5104
785.0	0.5172	0.5133	0.5121
804.8	0.5298	0.5291	0.5216
824.8	0.5548	0.5532	0.5410
844.8	0.5711	0.5703	0.5558
864.8	0.5799	0.5791	0.5618
884.9	0.5874	0.5851	0.5666
904.8	0.5905	0.5878	0.5693
925.0	0.5939	0.5916	0.5718
945.1	0.6103	0.5990	0.5751
965.0	0.6296	0.6253	0.5843
984.4	0.6449	0.6387	0.5903

Table IV. Specific Heat Capacity of Udimet Alloy 720

Temperature (K)	U720-AR $(\mathrm{J \cdot g^{-1} \cdot K^{-1}})$	U720-PT-1 $(\mathrm{J \cdot g^{-1} \cdot K^{-1}})$	U720-PT-2 $(\mathrm{J \cdot g^{-1} \cdot K^{-1}})$
113.5	0.2344	0.2301	0.2336
133.6	0.2848	0.2814	0.2845
153.5	0.3175	0.3147	0.3184
173.6	0.3450	0.3425	0.3458
193.7	0.3687	0.3654	0.3695
213.6	0.3864	0.3835	0.3876
233.6	0.3998	0.3967	0.4008
253.5	0.4116	0.4088	0.4126
273.5	0.4245	0.4216	0.4252
293.5	0.4348	0.4329	0.4351
313.5	0.4417	0.4357	0.4387
333.6	0.4485	0.4420	0.4453
344.2	0.4468	0.4454	0.4461
364.3	0.4521	0.4505	0.4513
384.2	0.4562	0.4549	0.4555
404.2	0.4605	0.4592	0.4598
424.4	0.4642	0.4630	0.4644
444.4	0.4687	0.4677	0.4683
464.3	0.4719	0.4709	0.4715
484.5	0.4758	0.4748	0.4753
504.5	0.4792	0.4784	0.4790
524.5	0.4813	0.4803	0.4809
544.6	0.4851	0.4837	0.4844
564.5	0.4874	0.4862	0.4866
584.5	0.4910	0.4893	0.4092
604.6	0.4931	0.4918	0.4923
624.6	0.4971	0.4956	0.4961
644.7	0.4991	0.4981	0.4987
664.7	0.5027	0.5007	0.5014
684.7	0.5050	0.5031	0.5039
704.7	0.5081	0.5065	0.5074
724.7	0.5105	0.5094	0.5101
744.9	0.5133	0.5120	0.5120
764.7	0.5163	0.5147	0.5155
785.0	0.5187	0.5174	0.5187
804.8	0.5209	0.5195	0.5210
824.8	0.5258	0.5234	0.5247
844.8	0.5395	0.5319	0.5332
864.8	0.5565	0.5467	0.5489
884.9	0.5709	0.5608	0.5628
904.8	0.5749	0.5674	0.5683
925.0	0.5787	0.5697	0.5710
945.1	0.5806	0.5735	0.5748
965.0	0.5902	0.5808	0.5816
984.4	0.5925	0.5809	0.5817

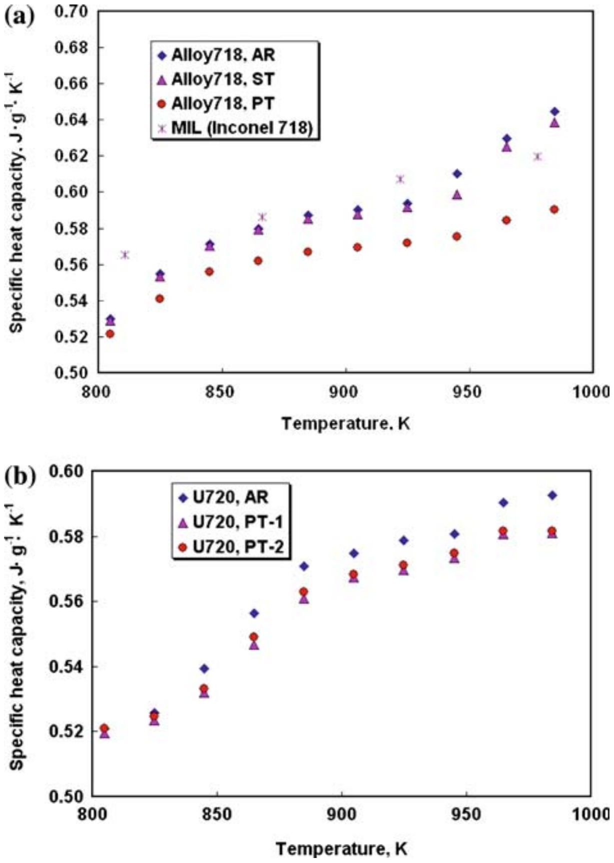

Fig. 4. Specific heat capacity of (a) Alloy-718 and (b) Udimet alloy 720 from 800 to 1000 K.

Udimet alloy 720:

$$
\begin{aligned}
C_p(\mathrm{J\cdot g^{-1}\cdot K^{-1}}) &= -0.30003 + 7.7512\times10^{-3}T - 3.5416\times10^{-5}T^2\\
&\quad +8.9519\times10^{-8}T^3 - 1.2772\times10^{-10}T^4\\
&\quad +9.6605\times10^{-14}T^5 - 3.0099\times10^{-17}T^6 \quad 100\,\mathrm{K} < T < 800\,\mathrm{K}.
\end{aligned}
$$

The polynomial fits agree well with the experimental data except at very low temperatures.

4. CONCLUSIONS

The specific heat capacity of nickel-based superalloys was measured by DSC. The temperature dependence of the specific heat capacity of the nickel-based alloys was determined over the temperature range from 100 to 1000 K. An anomalous increase in the specific heat capacity over the temperature range of 800 – 1000 K was attributed to precipitation. The specific heat capacity of the nickel-based alloys increases monotonically; however, above 800 K, it was strongly dependent on the heat treatment conditions, and was thought to be influenced by the precipitation phases (γ', γ'').

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