Structure-Property Correlation of Ni Superalloys manufactured by Laser Metal Deposition

Abstract:

Components made of Ni superalloys are installed, e.g., in hot parts of gas turbines. They withstand operating temperatures up to 1100 °C due to their high temperature strength and corrosion resistance. These properties are achieved upon heat treatment (HT), which yields a highly ordered two-phase system, referred to as γ- and γ'-phases, reducing plastic deformation by impeding dislocations. So far, worn components had to be replaced. Meanwhile, additive manufacturing techniques such as laser metal deposition (LMD) allow parts to be repaired locally, reducing maintenance costs. This type of material is prone to cracking, therefore understanding of the influence of microstructure on crack formation is crucial. Aside from mechanical properties, little was known about how microstructure affects macroscopic properties of Ni superalloys. Therefore, two goals of this work are (i) to characterize the microstructure from a mm- to nm-scale using electron microscopy techniques and (ii) to establish a structure-property correlation for magnetic and electrical properties.

Common materials such as $Ni_{60}Cr_{17}Co_8Al_7Ti_4Ta_1W_1Mo_1Nb_1$ (short $Ni_{60}Cr_{17}Al_7$, "IN738") were LMD grown on single crystalline $Ni_{60}Cr_{14}Co_9Al_8Ti_5Ta_1W_1Mo_1$ (short $Ni_{60}Cr_{14}Al_8$, "PWA1483") substrates for this purpose. Laser power, laser speed and mass flow were varied to study their influence on cracking. The 3 mm thick deposits, covering $10 \times 10\,\mathrm{mm}^2$ of the 10 mm thick substrate, were exposed to HT.

Electron backscatter diffraction (EBSD) yielded maps and pole figure texture plots of the polycrystalline $Ni_{60}Cr_{17}Al_7$ deposits to characterize the microstructure on a µm- to mm-scale. Average grain sizes between 75 µm and 196 µm correlate with crack densities between zero and $2.1\,\mathrm{mm}^{-2}$, which rise with increasing laser power and decreasing mass flow. Hot cracking occurs at large-angle grain boundaries (>15°). HT reduces grain size by about 10% and increases crack density by $0.3\,\mathrm{mm}^{-2}$.

Particular attention was given to microstructural changes in lattice parameters, chemical composition and phase fractions of the γ- and γ'-phase, since their precipitation changes physical properties. Therefore, microstructural analysis on a nm- to µm-scale was performed using a JEOL JEM-2200FS transmission electron microscope (TEM) with an in-column Ω-energy filter. The energy filter significantly improves imaging and diffraction. After LMD, γ'-phase precipitates 15 nm in size were already formed from the as-built γ-solid solution. After HT, the γ'-phase is about 500 nm in size and has a Ni_3Al crystal structure (space group $Pm\overline{3}m$). The chemically different γ-matrix has a Ni crystal structure ($Fm\overline{3}m$) with the same crystal orientation and nearly identical lattice parameter. This was determined by convergent beam electron diffraction (CBED) to be 0.361 nm for both phases.

To investigate plastic properties and crack initiation, TEM images recorded under defined two-beam diffraction conditions reveal dislocations and their interaction with precipitates. Grain boundary carbides cause superdislocations in γ' due to high internal strains and are therefore considered as crack initiators. The dislocation density of $3.0 \times 10^{10}\,\mathrm{cm}^{-2}$ decreases to $2.0 \times 10^{10}\,\mathrm{cm}^{-2}$ after HT. No stacking faults or twins were observed, although studies on medium entropy alloy NiCrCo suggest that stack-

ing fault energy (SFE) for the Ni superalloys studied is only about one-third of that of pure fcc Ni. Both the $Ni_{60}Cr_{17}Al_7$ deposits and the $Ni_{60}Cr_{14}Al_8$ substrate showed homogeneously dispersed granular TEM contrasts a few nm in size with a density of 10^{16}-$10^{17}\,cm^{-3}$. Ending half-planes and lattice distortions were associated with these contrasts by high-resolution TEM. Selected area electron diffraction (SAED) showed a superposition of diffraction patterns rotated 30° to each other.

Energy dispersive X-ray (EDX) spectroscopy in TEM of the γ- and γ'-phase, provided quantitative compositions from nm-sized volumes. Since physical properties are highly dependent on phase composition, the accuracy of the quantification was verified using newly calibrated Cliff-Lorimer k-factors on both a JEOL JEM-2200FS at Siemens in Munich and a FEI Tecnai F20 at TU Wien. In $Ni_{60}Cr_{17}Al_7$ deposits, the compositions of γ and γ' were determined to be $Ni_{54}Cr_{27}Al_4$ and $Ni_{70}Cr_3Al_{12}$. The $Ni_{60}Cr_{14}Al_8$ substrates yielded γ- and γ'-phase compositions of $Ni_{54}Cr_{24}Al_5$ and $Ni_{68}Cr_3Al_{12}$. From the nominal composition and the obtained phase compositions, the volume fraction for the γ'-phase established to range from 39% to 45%.

Measurements of magnetization in a superconductive quantum interference device (SQUID) and magnetoresistivity were carried out at TU Wien. The measurement range was chosen from 2 K to room temperature in magnetic fields up to 9 T to study the long-range magnetic order and possible superconductivity. The Ni superalloys are paramagnetic at 5 K and 7 T. HT reduced magnetization from 5.3×10^3 A/m to 4.3×10^3 A/m and magnetic susceptibility from 1.5×10^{-3} to 1.1×10^{-3}, due to formation of the Ni_3Al-based γ'-phase. Based on TEM microstructural analysis, Brillouin functions were calculated with $0.62\,\mu_B$, $0.075\,\mu_B$, and $1.72\,\mu_B$ for magnetic moments of Ni, Ni in Ni_3Al, and Co atoms. Only about 4% of the magnetic moments are aligned at 5 K and 7 T. The depinning factor does not depend on the magnetic field, only on the temperature. Co accounts for most of the magnetic moment. Magnetoresistivity was 0.3 µΩcm at 4 K and 9 T and it correlated linearly with magnetization. γ'-phase precipitation caused both residual electrical resistivity and nanoindentation Young's modulus to increase from 126 µΩcm to 137 µΩcm and from 207 GPa to 224 GPa, respectively.

The studies show that LMD parameters have a crucial influence on microstructure and crack density. Cracks occur only along grain boundaries, their density correlates to increasing grain size and heat input. Cracking can be initiated by locally induced stress from carbides. It is possible to obtain 3 mm thick crack-free deposits using reduced laser power and increased mass flow. HT is established as a key processing step for physical properties due to γ'-phase precipitation. The level of γ'-formation can be monitored by magnetization and magnetoresistivity measurements due to the investigated dependence of macroscopic properties on microstructure. This provides the potential for a novel non-destructive testing method to draw conclusions about the microstructural transformation of Ni superalloys from macroscopic properties.