Qualification of electron-beam welded joints between copper and stainless steel for cryogenic application

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Abstract. Joints between copper and stainless steel are commonly applied in cryogenic systems. A relatively new and increasingly important method to combine these materials is electron-beam (EB) welding. Typically, welds in cryogenic applications need to withstand a temperature range from 300 K down to 4 K, and pressures of several MPa. However, few data are available for classifying EB welds between OFHC copper and 316L stainless steel. A broad test program was conducted in order to qualify this kind of weld. The experiments started with the measurement of the hardness in the weld area. To verify the leak-tightness of the joints, integral helium leak tests at operating pressures of 16 MPa were carried out at room- and at liquid nitrogen temperature. The tests were followed by destructive tensile tests at room temperature, at liquid nitrogen and at liquid helium temperatures, yielding information on the yield strength and the ultimate tensile strength of the welds at these temperatures. Moreover, nondestructive tensile tests up to the yield strength, i.e. the range in which the weld can be stressed during operation, were performed. Also, the behavior of the weld upon temperature fluctuations between room- and liquid nitrogen temperature was tested. The results of the qualification indicate that EB welded joints between OFHC copper and 316L stainless steel are reliable and present an interesting alternative to other technologies such as vacuum brazing or friction welding.

Keywords: Electron-beam welding, copper, stainless steel, cryogenic application, 316L, mechanical test, hardness, liquid nitrogen, liquid helium

1. Introduction
The joints between oxygen-free high conductivity copper (OFHC) and 316L play an important role for cryogenic application. A possibility to join these materials is electron-beam (EB) welding. Upon the impact of the electron beam on the materials, the kinetic energy of the electrons is transformed into heat and causes the materials to melt. To prevent dispersion of the electron beam and to avoid oxidation of the metal, the welding is mostly performed in a vacuum. One of the many advantages of this method are the deep and narrow welds as a result of the high energy density. This method is often used in aerospace industries since EB welding is well suitable for titanium and titanium alloys, which are the most widely used materials in aerospace industries [1].
EB welding between 316L and OFHC will be used for a new cryogenic mass flow meter for the determination of mass flow rates in gases and liquids. The method of this new mass flow meter is explained in [2]. A technology transfer project together with WEKA AG was started in order to develop this new mass flow meter for a working range of 300 K down to 4 K and with pressures up to 50 bar.

As few data are available for classifying EB welds between 316L and OFHC, specially designed samples were produced and tested between 300 K and 4 K, and with high pressures. The geometries were close to the original design of the mass flow meter. Figure 1 shows a technical drawing and pictures of the samples used for the tests.

In the first chapter, this paper discusses the test program. Hence, the results of the test program are presented and discussed. Finally, conclusions on the qualification of the EB weld for cryogenic application are made.

2. Test program and methods
The test program shown in figure 2 was created and conducted under consideration of the special conditions for cryogenic application. First, a hardness test was conducted to check the influence of the welding on the microstructure of the materials in the area of the weld. The Vickers HV0.2 hardness test\(^1\) was conducted according to ISO 6507-1 [3]. Second, helium pressure and leak tests at room temperature (RT, 300 K) were conducted with the aim to prove the tightness and pressure resistance of the weld. Helium pressure and leak tests at RT and at cryogenic temperature were conducted according to EN 1779 [4]. The integral leak rate for the whole sample (shown in figure 1(b)) was measured and helium was used as test gas\(^2\). Third, destructive tensile tests at RT, at liquid nitrogen (LN\(_2\)) temperature (77 K), and at liquid helium (LHe) temperature (4 K) were performed to reveal information on the EB weld such as yield strength \(\sigma_y\) and ultimate tensile strength \(\sigma_u\).

\(^1\) The hardness test was performed with the NEXUS 4000 hardness tester from the INNOVATEST Europe BV.
\(^2\) The leaking helium was detected by a leak indicator of the type Leybold UL 200 dry with scroll pump SC 5 D from the Oerlikon Leybold Vacuum GmbH.
Figure 2. Test program per sample in the order of the performed tests. For a better overview, the samples were numbered (e.g. A1, A2, ...).

Since plastic deformation of the sample is avoided during operation, nondestructive tensile tests up to yield strength were conducted, also at RT, at 77 K, and at 4 K in order to see if the weld will be influenced. Both destructive and nondestructive tensile tests, were performed in the CryoMak laboratory at the Institute for Technical Physics (ITEP) whose facilities are explained in [5]. The tests were conducted according to ISO 6892-1 [6] at RT, ISO 15579 [7] at 77 K, and ISO 19819 [8] at 4 K. The deformation during the tests was measured by two extensometers and it was intended to have an initial gauge length of $L_0 = 22$ mm (see figure 3). Therefore, the measured deformation includes the deformation of the different materials in the various areas (316L, OFHC, weld). Since the EB weld is to be qualified for cryogenic application, it is important to know how the weld reacts to temperature fluctuations. Therefore, a temperature cycling test was conducted. Two samples (sample design is shown in figure 1(b)) were put into a box filled with LN$_2$, where they were cooled down to 77 K. Afterwards, the samples were reheated to RT by a dryer. This cycle was repeated ten times. Subsequent to the nondestructive tensile tests and the temperature cycling test, helium pressure and leak tests were performed to prove that the weld was not damaged by the previous tests. These pressure and leak tests took place at cryogenic temperature.

Figure 3. For the tensile tests, two extensometers are mounted on the sample with an initial gauge length of $L_0 = 22$ mm.
3. Results and discussion

3.1. Hardness test

Figure 4 shows the results of the measured hardness for the indentations of line 4. As can be seen, the hardness was not constant in the weld region. This is the result of an inconsistent microstructure in this area, which was caused by the welding and is a usual behavior. The transparent gray zone in figure 4 was found to have a width between 100 and 150 µm in 316L and between 75 and 100 µm in OFHC. In this area, the welding also changed the microstructure of the materials. This zone is wider in 316L because of its lower thermal conductivity compared to OFHC. Next to this gray zone, the hardness was found to be constant on both sides and corresponds to literature values of the used materials. Therefore, it could be shown the welding had no influence in this part. The same results were found for the other lines.

Figure 4. The measured hardness of each indentation of line 4. The dashed white line shows the border between the weld and the pure materials on both sides and the transparent gray zone indicates the area, where the welding had an influence on the microstructure of the pure materials.
3.2. Helium pressure and leak tests
The samples were always pressurized between 130 bar and 160 bar, which was the maximum available pressure depending on the available pressure of the helium bottle and the weld withstood this high pressure.

Several pressure and leak tests at RT were conducted and the measured leak rate was found to be in the range of $\leq 3.2 \cdot 10^{-9} \text{mbar l s}^{-1}$ and $\leq 1 \cdot 10^{-10} \text{mbar l s}^{-1}$ for all tests. Moreover, several pressure and leak tests at about 80 K were performed and the leak rate was measured in the range of $\leq 7 \cdot 10^{-10} \text{mbar l s}^{-1}$ and $\leq 1 \cdot 10^{-10} \text{mbar l s}^{-1}$ for all tests. These results were in the minimum detectable range of the used test setup. Also, external influences had an impact on the results and consequently the background determines the result and therefore the results were like expected. The results of the pressure and leak tests demonstrated that EB welding can join materials without leakages and with sufficient tightness even at high pressures and cryogenic temperatures.

3.3. Temperature cycling test
An external change was not apparent after conducting the temperature cycling test and also the subsequent pressure and leak test showed no difference to previous pressure and leak tests. Consequently, the EB weld seams to be resistant against temperature fluctuations in the temperature range between RT and 77 K.

3.4. Destructive tensile tests
Figure 5 shows the resulting stress-strain curves for the destructive tensile tests at different temperatures. Since the fracture caused by the destructive tensile tests was in the weld for all tests, the stress was measured with an assumed cross-sectional area $A_{CS}$ in the part of the weld ($A_{CS,weld} = 44 \text{mm}^2$). The breaking strain decreases with lower temperature since the materials are more sensitive to fail at lower temperature. Moreover, the 0.2 % proof stress $\sigma_{0.2}$ is higher with a lower temperature as well as the yield strength $\sigma_y$. $\sigma_y$ is in the range of 40-50 MPa and consequently the weld should not be stressed higher during the real operation.

Figure 5. Stress-strain curves for the destructive tensile tests at RT, at 77 K, and at 4 K. (a) shows the curves in total and (b) shows a zoom into the elastic zone. The curve for the test at RT was measured with sample D1, for the test at 77 K with sample A2, and for the test at 4 K with sample E2.
σ_{0.2} of the used OFHC is in the range of the measured σ_{0.2} of the weld. Therefore, the OFHC is the weak point of the joint. Since the slope of the three curves in the elastic zone is in the same range, the modulus of elasticity E hardly changes with temperature. In total, the shape of the curves show the expected results.

The measured values for the mechanical parameters and their uncertainties, calculated according to the Guide to the expression of uncertainty in measurement [9], are shown in table 1.

### Table 1. Results of the destructive tensile tests at RT, at 77 K, and at 4 K

| Parameter | unit | RT     | 77 K   | 4 K    |
|-----------|------|--------|--------|--------|
| σ_u       | MPa  | 500 ± 1.5 | 600 ± 1 | 565 ± 1.25 |
| A         | %    | 9.35 | 7.75   | 5.8    |
| σ_y       | MPa  | 40 ± 1.5 | 45 ± 1  | 50 ± 1.25 |
| σ_{0.2}   | MPa  | 105 ± 1  | 114 ± 1 | 126 ± 3  |
| E         | GPa  | 260 ± 35 | 270 ± 22 | 260 ± 144 |

3.5. Nondestructive tensile tests

The destructive tensile tests indicated a yield strength of about σ_y = 45 MPa, giving the operational limit of the weld. In order to see if stress below σ_y already influences the weld, nondestructive tensile tests at RT, at 77 K, and at 4 K up to σ_y have been conducted. The resulting stress-strain curves can be seen in figure 6.

Since it was tested in the lower force range of the machine, the maximum stress that occurred on the weld was difficult to control. Consequently, the weld was stressed into the plastic region for the test at RT and 77 K and a permanent deformation occurred. No plastic deformation occurred for the test at 4 K (see figure 6(e) and (f)). The scattering of the measuring values is wider for the test at 4 K because of the thermal convection at low temperature, but the resulting uncertainty is included in the results. As the curves show, the slope in the elastic zone varies for tests at same temperatures, especially at 77 K (see figure 6(c) and (d)). The reason is that the positioning across the EB weld varies slightly leading to different fractions of 316L and OFHC across the gauge length. Furthermore, slight differences in the shape of the weld - differences in A_weld - influence on the results and effect a different slope in the elastic zone.

Subsequent to the nondestructive tensile tests at different temperatures, helium pressure and leak tests were performed. The results of these tests showed that the nondestructive tensile tests did not damage the weld since the measured leak rate was found to be in the range of the previous pressure and leak tests (between 10^{-10} mbar l s^{-1} and 10^{-9} mbar l s^{-1}). In consequence, the EB weld is resistant against stresses in the elastic region.
4. Conclusion
The performed test program with the hardness test, the helium pressure and leak tests, the destructive- and nondestructive tensile tests and the temperature cycling test yields the desired results. The EB weld was resistant against pressures of about 160 bar and also the weld was found to be leak-tight with measured leak rates smaller than $10^{-9}$ mbar l s$^{-1}$. The elastic region...
was measured between 40 MPa and 50 MPa for all performed tensile tests at RT, 77 K, and 4 K and the temperature cycling from RT to 77 K did not damage the weld. Therefore, EB welding is a suitable method for cryogenic application.

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