Determination of fracture toughness K_{Ic} of small hard particles embedded in a soft matrix using microindention and microscopy

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Fracture toughness is an extremely important property of a structural material. It namely determines the resistance of a material against instable crack propagation, and consequently affects the fracture safety. One of the methods for determination of fracture toughness of brittle materials, such as ceramics, intermetallic phases and quasicrystals is the indentation technique [1, 2]. The cracks, which initiate at indentation corners (e.g. by the application of Vickers hardness measurement) and for which verifies the condition $0.25 \leq l/d \leq 2$ are known as Palmqvist cracks (Fig. 1). Their length l is directly correlated with the plane strain fracture toughness K_{Ic} [3]:

$$K_{IC} = 0{,}035 \cdot \Phi^{-3/5} \cdot HV \cdot (\frac{HV}{E})^{-2/5} \cdot (\frac{d/2}{\sqrt{l}}) \qquad (1)$$

where Φ is a constraint factor (≈ 3), HV is a measured Vickers microhardness, E is Young's modulus and d is a diagonal of the indent ($d = 2a$).

Determination of fracture toughness is not limited to bulk specimens and thin films only, but determination of fracture toughness of individual phases in multiphase microstructures is rather challenging. This is particularly true for tiny particles having small dimensions in both lateral and depth directions. In this case it is very difficult to comply with corresponding standards for valid measurements. This difficulty appeared in investigated Al-Mn-Be alloys. The microstructure of the alloys consisted of a quasicrystalline i-phase, hexagonal $Al_{10}Mn_3$ and cubic Be_4AlMn embedded in Al-rich matrix; hard dispersed phases in a tough matrix. Since in the open literature no information was found regarding the procedures for performing reliable microindentation measurements and determination of fracture toughness on small particles, it was our goal to find the most important parameters influencing the results of the measurements.

Scanning electron microscopy (SEM) with focused ion beam (FIB) has an important role for correct selection of the individual results and for interpretation of unusual shapes of indentation curves (FIB), determination of thickness of an investigated particle (FIB), measurement of Palmqvist crack lengths (SEM), verification whether cracks, crack networks or other phases are present below an indent (FIB).

The selection of measurement for evaluation of the result were made with microscopy analysis, from the shape of indentation curve and measurement results. The measurement was excluded if one of the following criteria was satisfied:

- the hardness values or modulus elasticity much smaller then average values;
- the indent was too close to the edge of a particle;
- the indentation depth was close or larger that thickness of a particle;
- the crack stopped at a phase boundary or even proceeded into neighbouring phase;
- the crack length did not satisfy the condition $0.25 \leq l/d \leq 2$.

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The minimum size of a particle strongly depends on a minimum load for nucleating a crack. In general the critical size of a particle is proportional to the minimum load, e. g. higher is the minimum load, larger should be the particle size. It was found out that the minimum lateral size of a particle *D* should be at least should be at least 3*d*, to obtain reproducible results.

It can be concluded that fracture toughness of small dispersed phases can be determined in the case when the particle size exceeds the critical value which depends on hardness, modul of elasticity and in the greatest extend, minimum force for creating crack.

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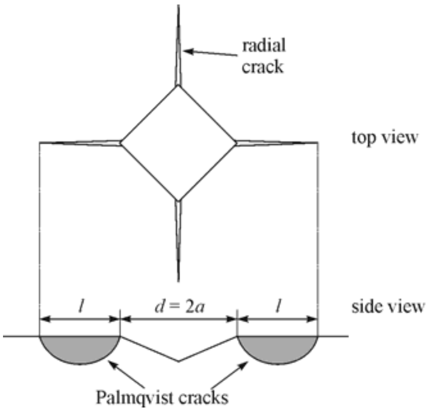
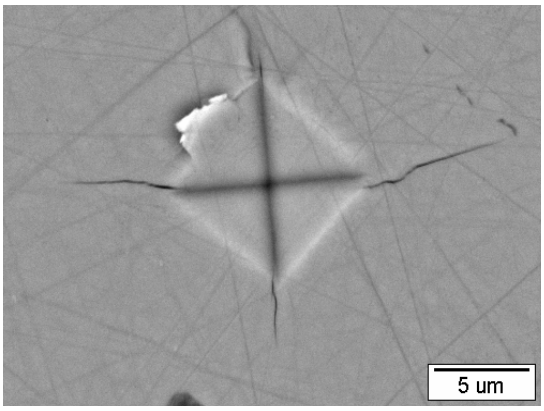
Table 1: Mechanical properties of investigated phases in Al-Mn-Be alloys

phase	modulus of elasticity *E* / GPa at 20 mN	Vickers hardness / GPa at 20 mN	minimum force for crack nucleation / mN	fracture toughness K_{Ic} / MPa $m^{1/2}$
i-phase	161 ± 9	22.53 ± 2	30	1.01 ± 0.2
$Al_{10}Mn_3$	175 ± 6	8.08 ± 0.3	100	0.94 ± 0.09
Be_4AlMn	253 ± 8	13.37 ± 0.2	50	1.32 ± 0.2





a) b)

Figure 1: a) Scheme of the Palmqvist crack, b) SEM micrograph of an indent with Palmqvist cracks

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