FATIGUE CRACK BEHAVIOR OF ULTRA FINE GRAIN PURE METALS PRODUCED VIA SEVERE PLASTIC DEFORMATION

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Abstract
The fatigue crack behavior of metals is strongly governed by the grain size variation. As for tensile strength, the fatigue limit increases with decreasing grain size up to the microcrystalline regime. A different behavior in terms of mechanical properties has been demonstrated in many papers for metals with ultra fine (< 1 μm) and nanocrystalline (< 100 nm) grain size, in particular for the yield stress value and fatigue crack initiation and growth. The fatigue crack propagation of pure titanium, pure nichel and copper with ultra fine grain structure has been analyzed in the present paper. In particular, the fatigue crack initiation phase and growth of these materials in the form of thin plates was studied over a broad range of stress levels and life cycles.

Keywords: ultra fine grain, pure metals, severe plastic deformation, fatigue crack growth.

Introduction
The mechanical properties of metals are strongly influenced by grain size. In the recent years a strong attention has been devoted to the increment of monothonic resistance and fatigue life on nanocrystalline and ultra-fine grain size materials [1-3]. In particular, the effect of severe plastic deformations on the metal grain refinement and the consequent improvement of mechanical properties has been widely studied [4, 5].
Many papers have been published regarding the hardness and yield strength increments in materials refined via severe plastic deformation [6-12], but many aspects are to be analyzed to better understand their resistance against cyclic loading. Some authors analyzed the strain controlled behavior under fatigue load of pure Cu, brass and Aluminium specimens, demonstrating a decrease in fatigue resistance of such materials produced by equa-channel-angular pressing (ECAP), especially in the range of intermediate and high plastic strain ranges [13-15]. Although the static tensile strength of ECAP copper is 1.8 times higher than conventional material, Han et al. showed no increase in fatigue limit stresses to occur [16]. On the contrary, a relevant improvement in fatigue endurance has been checked for aluminium alloys [17-20]. While other authors demonstrated the resistance increase of pure Ti processed via ECAP under stress and strain controlled fatigue loading [21, 22]. Despite all these information achieved with stress and strain controlled fatigue testing, few papers are to be found in literature about the fatigue crack initiation and propagation in materials treated via severe plastic deformation and presenting ultrafine grain microstructure [3, 18, 23].
The aim of the present paper is to investigate on the fatigue crack behavior under stress controlled tests of pure Ni, Ti and Cu produced via equa-channel-angular pressing.

Experimental procedure
ultra-fine grain microstructure achieved with ECAP
The ultra fine grain materials analyzed in the present work were:

- pure Ti grade 2 produced by ECAP with 8 passes route Bc by employing a 90° corner channel at the temperature of 425 °C; the extruded billets measured 45 mm in diameter and 100 mm in length;

- pure Ni produced by ECAP with 8 passes route Bc by employing a 90° corner channel at the temperature of 750 °C; the extruded billets measured 45 mm in diameter and 100 mm in length;
- pure Cu produced by ECAP with 4 passes route Bc by employing a 90° corner channel at room temperature; the extruded billets measured 45 mm in diameter and 100 mm in length.

The materials microstructure was studied with TEM observations; flat disks obtained in the extrusion direction were mechanically polished up to a thickness of 40 nm and then twin jet polished in methanol+ 30% HNO_3 solution in dry ice at -60°C, finally the samples were alsop polished by ion milling in liquid nitrogen for 2 hours. The observations were performed by employing a JEOL 2011FX TEM.

Crack growth testing and fracture analysis

For the fatigue crack propagation tests, different disks of 2 mm thickness were cut perpendicularly to the extrusion direction and different specimens in C(T) (Fig 1) configuration were obtained by EDM. The pre-cracking line cut has not been executed on the specimens.

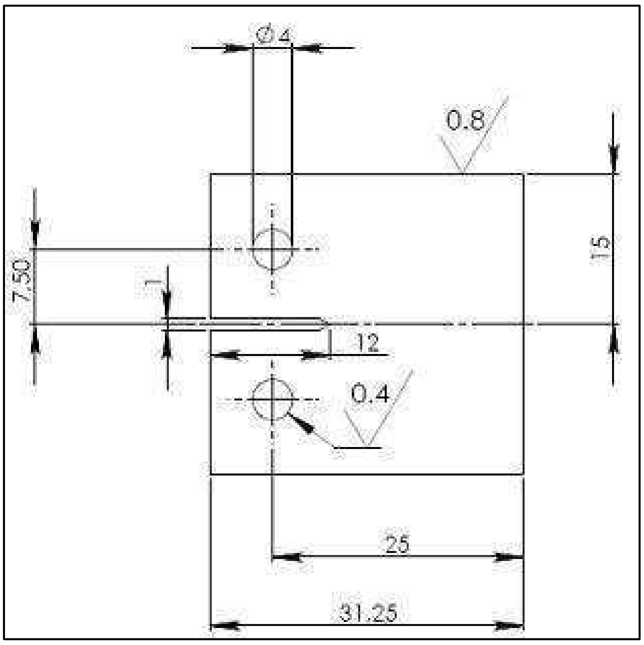

Figure 1: Geometry of the specimens used for the crack initiation and growth tests, all the dimensions are in mm.

The fatigue crack propagation tests were performed with an axial resonant fatigue machine of type Rumul 50 kN, with load ratio R=0.25 at 60 Hz. The crack advances were monitored optically by using a Watek 505EX CCD high speed camera with 1000 frames/s and accurate measurements of the crack line with selected images of tensioned specimens (Fig. 2). The fatigue tests were conducted until complete specimen failure and the timed data of force, displacement and crack length were collected and elaborated.

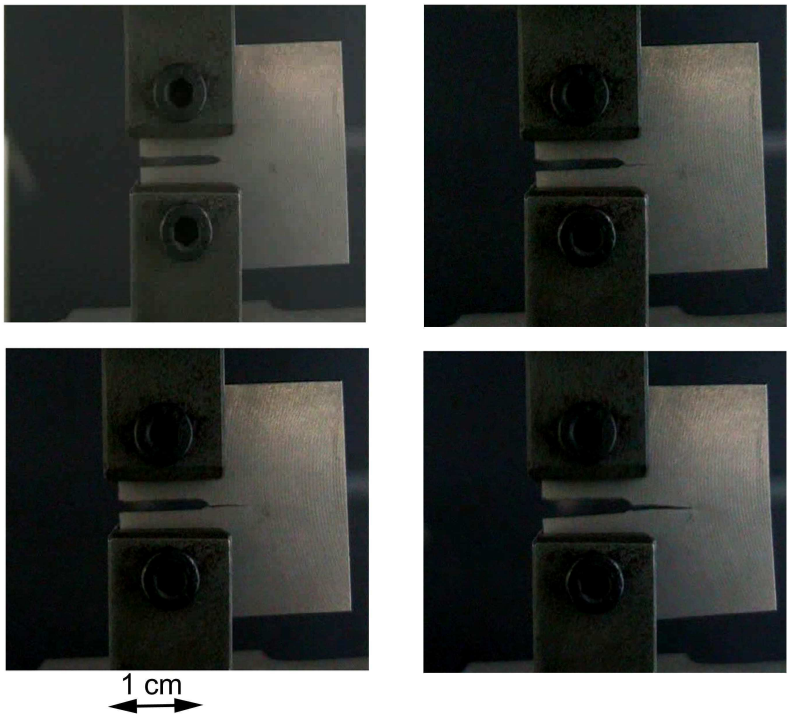

Figure 2: Example of the crack behaviour in the ECAP Ti sample.

Results and discussion

In Figure 3, the TEM micrographs of the studied materials after ECAP are shown.

All the microstructures revealed a mean grain size around 300 nm with a characteristic shear band formation for the ECAP copper.

In the present study, the fatigue crack growth response of UFG pure Ti, Ni and Cu plates produced by severe plastic deformation was analyzed from threshold to final failure. The studied metals yield strength is reported in Table I.

The fatigue crack propagation data are expressed in terms of stress intensity ranges ΔK [MPa $m^{0.5}$] for Ti and Ni specimens in fig. 4 a-b, where the base microcrystalline metal data are also reported to compare the results; the maximum applied stresses during the tests are reported in Table II. The crack propagation curves were achieved taking into account the evaluation of the shape factor described in previous studies.

For such metals it can be observed the grain refinement due produces a consistent reduction of ΔK threshold value and a consequent increase of crack propagation rates for a given stress state; the curve shape is kept identical and a large data shift effect is measured for UFG specimens. This behavior is due to the different crack paths observed in these materials with different grain size and by the cyclic hardening effect that titanium and nickel experienced in the experiments.

Table I: Yield stress of the studied materials in the as-received condition and after severe plastic deformation.

Material	Yield stress (MPa)
UFG-Ti	650
mc-Ti	415
UFG-Ni	490
mc-Ni	380
UFG-Cu	340
mc-Cu	150

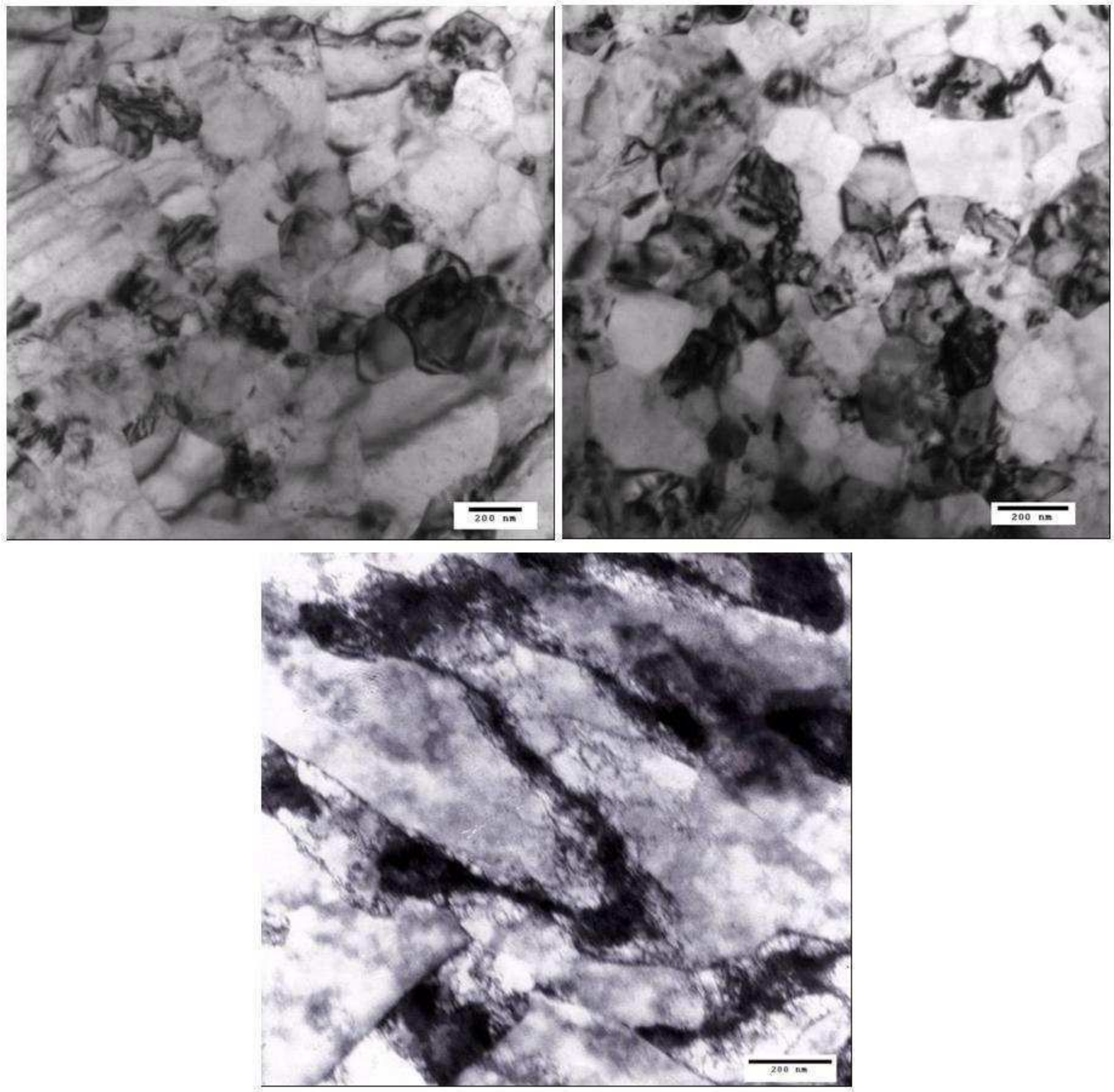

Figure 3: Microstructure of the studied metals after severe plastic deformation; Ti a), Ni b), Cu c).

Table II: Maximum stresses applied during the fatigue crack tests, the load ratio was R=0.25 for all the specimens.

Material	Maximum stress (MPa)
UFG-Ti	510
mc-Ti	320
UFG-Ni	380
mc-Ni	300
UFG-Cu	260
mc-Cu	120

Such behavior has been also observed in nanocrystalline Nickel produced by electrodeposition [24]. As shown in figure 5, the crack path of the ultra-fine Ti appears much more flat with respect to the microcrystalline counterpart.
A completely different behavior was observed for the UFG pure copper specimens (Fig. 6).

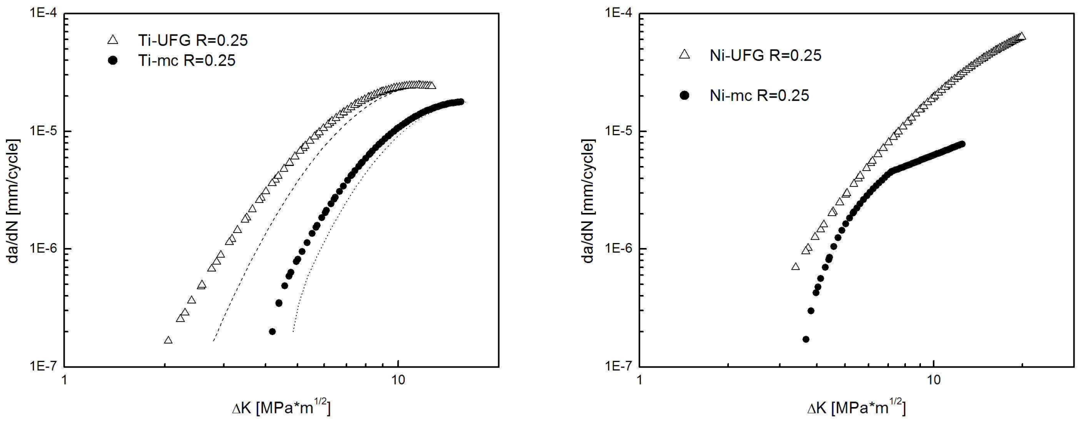

Figure 3: Variation of the fatigue crack growth rate da/dN vs. ΔK for the ufg Ti a) and Ni b) at a load ratio R=0.25.

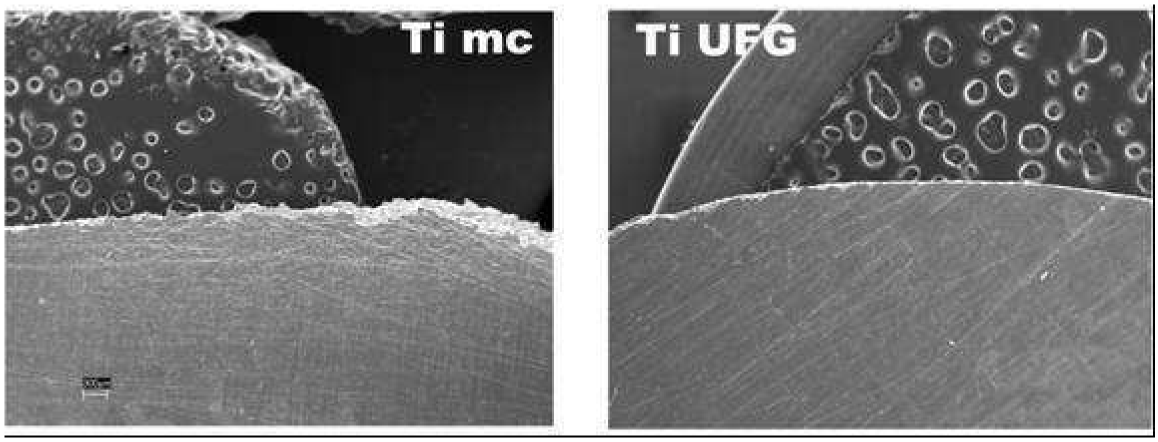

Figure 5: Different crack paths observed for the UFG Ti and the microcrystalline counterpart.

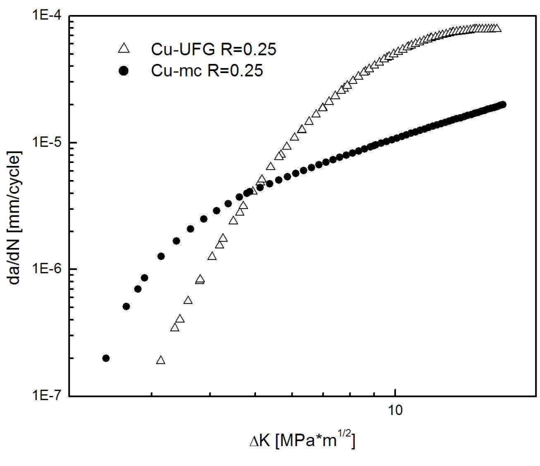

Figure 6: Variation of the fatigue crack growth rate da/dN vs. ΔK for the ufg Cu at a load ratio R=0.25.

In such case, the material subjected to ECAP processes shows an higher susceptibility to crack initiation and a faster crack growth rate with respect to the microcrystalline material, especially when the loading cycles are increasing.

Figure 7: Comparison of the crack paths of the UFG metals analyzed in the present study.

Such behavior has been already assessed for severe plastic deformed copper which exhibits a large cyclic softening effect under repeated loading. Also in this case one of the factors affecting the different cracks growth behavior are the different crack paths (Fig. 7), which have been observed. For the UFG Cu and Ni tests, the paths are typically much more of ductile behavior with respect to the UFG Ti.

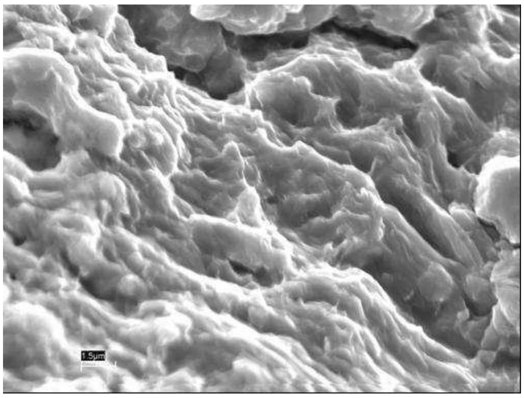

Figure 8: Fracture surface aspect of the UFG Ti.

The fracture surface aspect of ECAP titanium specimen is shown in fig, 8; it is characterized by local ductility filaments, proper of stable crack propagation.

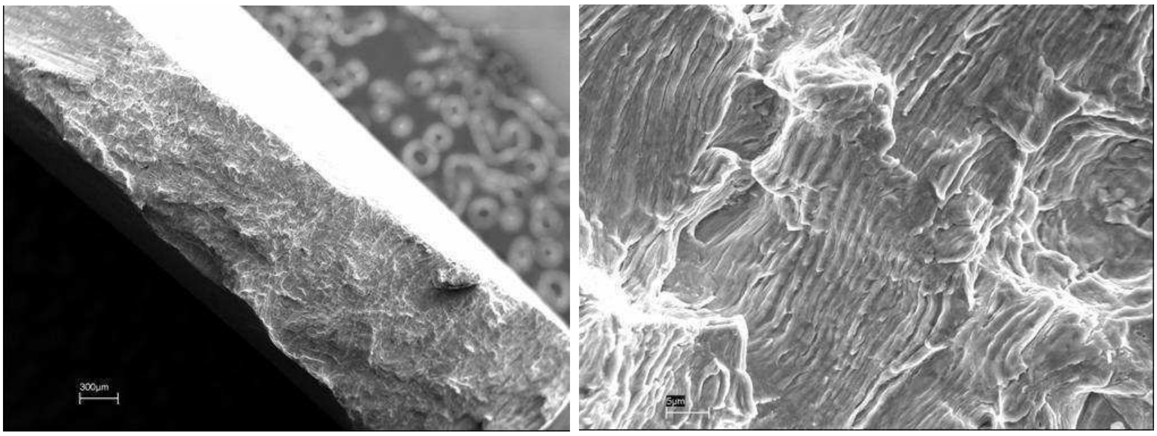

Figure 9: Fracture surface of the UFG Ni revealing macroscopic ductility a) and very fine fatigue striations b).

The UFG Ni showed instead macroscopic ductility (Fig. 9a) and fine striations on all the ruptured surface (Fig. 9b). Such macroscopic ductility is produced in several ways, depending on the observation spot (Fig. 10a) and local fine dimples (Fig. 10b) were detected for the UFG Cu.

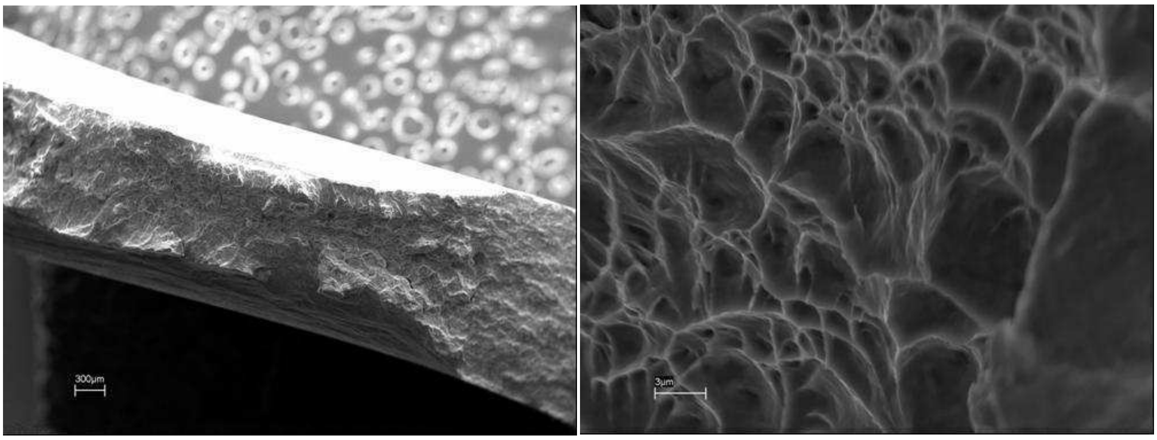

Figure 9: Macroscopic ductility revealed by the UFG Cu a) accompanied with a broad population of very fine dimples observed on the fracture surface b).

Conclusions

The fatigue crack behavior of pure Ti, Ni and Cu produced via equa-channel-angular pressing was analyzed and the results presented in the paper. The severe plastic deformation produced an increase in tensile strength for all the materials. For the pure Ti and Ni it was observed that the grain refinement due to the severe plastic deformation produces a decrease in ΔK threshold and an increase in crack propagation rate. On the contrary, the pure Cu revealed an higher susceptibility to crack initiation and a faster crack growth rate respect to the microcrystalline material especially increasing the loading cycles.

Acknowledgments

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