Small punch testing of Fe-Al based alloys with Ti and Nb additions

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Abstract. Three Fe-Al-based alloys, a binary with 22 at. % of Al, a ternary with 22 at. % of Al and 7 at. % of Ti and a quaternary with 22 at. % of Al, 4 at. % of Ti and 4 at. % of Nb prepared by arc melting to small button type ingots were studied by small punch test and small punch creep test in order to obtain the high temperature tensile and creep properties. Evaluation of the results shows a significantly improved strength at high temperatures and creep resistance of the ternary and quaternary alloys compared to the binary alloy. The observation of the punched discs fracture surfaces related to the initial microstructure also helps to better understanding of the deformation and fracture behavior of these alloys at high temperatures.

1 Introduction
The iron aluminides represent a group of materials with unique properties such as very good corrosion and chemical resistance, attractive strength to weight ratio and low cost if compared to more expensive Ni-based alloys or superalloys. A successful example of recent replacement of engine part originally produced from Ni-based superalloy can be found in [1]. The iron aluminides have also interesting magnetic properties. Recent investigations devoted to the structural and physical properties of the parent Fe-20 at. % Al alloy modified by Ti and Nb at the expense of Fe point to different microstructures of Ti+Nb alloyed samples compared to samples with only Ti addition [2]. Moreover, a huge increase in saturation magnetization of the sample with 4 at. % Ti + 4 at. % Nb compared to sample with 8 at. % Ti (87.5%) is unresolved and represents an interesting topic for the next experimental and theoretical investigations [3]. Aim of this study is to test and compare the high temperature mechanical properties (tensile and creep) of Fe-Al alloys with 3 different compositions using the cost-effective small scale testing. A detailed comparison of the first two alloys with the data found in the literature was done in [4].

2 Materials and methods
The studied alloys were prepared from high-purity elements (Fe – 99.95%, Ti, Nb – 99.8%, and Al – 99.95%) using arc melting in MAM-1 furnace (Buehler GmbH) into a button-type ingots. They were four times re-melted to guarantee a good chemical homogeneity. The chemical composition of the three studied alloys is given in Table 1.
Small punch tests were performed in two loading modes: (i) small punch creep (SPC) test at constant force under which the punch penetrates through the specimen and the time dependence of the punch displacement is measured; (ii) small punch test (SPT) at constant rate of punch displacement and the necessary force is measured. Both tests are run up to the rupture of the specimen. A constant load cantilever creep machine was adapted for testing. During the test, punch is pushed against a specimen supported by a 4-mm diameter receiving die (lower die). Punch tip consists of a precise ceramic ball made of FRIALIT F99.7, 2.5 mm in diameter. The disc specimen of diameter $D_S = 8$ mm and thickness $h_0 = 0.5$ mm is clamped by an upper die. Central deflection is measured as the difference in the positions of the punch and lower die, using a linear variable differential transformer and is continuously recorded with a PC. The force acting on the specimen is measured using the load cell. All tests are performed in a protective atmosphere of purified argon. Temperature is measured by thermocouple placed in the receiving die in the proximity of specimen. The technique is described in more detail elsewhere [5, 6].

The specimens for small punch testing were prepared by machining cylinders 8 mm in diameter. The cylinders were subsequently cut to slices 1.2 mm thick using electro discharge machining. The slices were ground carefully from both sides equally and finally polished to 1200 grit. The final thickness $h_0 = 0.500 \pm 0.005$ mm was measured by using a micrometer with a resolution of 1 μm.

The microstructural and fractographic observations were done using a scanning electron microscope Tescan Lyra3 XMU. Morphology studies and phase analyses were carried out with the transmission electron microscope JEM 2100F (Schottky cathode, using a 200 kV electron beam). TEM foils of as prepared alloys were prepared by grinding down to a grit size of P1200 and finished using an ion milling device (PIPS).

### 3 Results and discussion

#### 3.1 Microstructure

The polycrystalline microstructures of the samples were checked by scanning electron microscopy (SEM) and analyzed using transmission electron microscopy (TEM). The morphologies of the alloys in the “as prepared” state were substantially different. The polished cross section microstructures of three studied alloys in the as prepared state are shown in Fig. 1. The irregular larger grains are observed at the Ti-0 sample in Fig. 1a. A finer-grained microstructure was formed in the Ti-7 alloy as seen in Fig. 1b. The Ti-4-Nb-4 alloy microstructure in Fig. 1c shows a matrix formed by primary dendrites with eutectic similar to the Fe-Al-based alloy in [1]. The chemical analysis showed a low concentration of Nb in the matrix and high in the eutectic. TEM images of the sample Ti-0 showed nanocrystalline morphology (Fig 2a). TEM analysis of the sample Ti-7 revealed cuboids of the off-stoichiometric Heusler Fe:TiAl phase (crystal lattice L2₁) with the side lengths of 50 nm embedded in the bcc Fe-Al-(Ti) matrix (crystal lattice A2) (Fig 2b). TEM analysis of the sample Ti-4-Nb-4 shown in Fig. 2c from the dendrites that are Nb depleted (dark areas in SEM in Fig. 1c) using the selective electron diffraction also points to a superstructure but needs to be further evaluated. It seems that

| Alloy | Composition | Fe   | Al   | Ti   | Nb   |
|------|-------------|------|------|------|------|
| Ti-0 | intended    | 78   | 22   | 0    | 0    |
|      | analyzed    | 77.1±0.2 | 22.9±0.2 |      |      |
| Ti-7 | intended    | 71   | 22   | 7    | 0    |
|      | analyzed    | 70.2±0.2 | 22.6±0.2 | 7.2±0.1 |      |
| Ti-4-Nb-4 | intended | 70   | 22   | 4    | 4    |
|        | analyzed   | 69.5±0.2 | 22.5±0.2 | 4.1±0.1 | 3.9±0.1 |
addition of some alloying elements like Ti can lead to more regular substructure within the polycrystalline grains that might resemble the superstructure of the Ni-based superalloys.

![SEM micrographs](image1.png)  
**Fig. 1.** SEM micrographs of “as prepared” state of samples SEM back-scattered electrons a) Ti-0, b) Ti-7, c) Ti-4-Nb-4.

![TEM micrographs](image2.png)  
**Fig. 2.** TEM micrographs of “as prepared” state of samples: a) a nanocrystalline subgrain morphology of the alloy Ti-0 [4]; b) cuboid regions with the side lengths of ~50 nm of the alloy Ti-7 [4] c) morphology of the Nb depleted area of the alloy Ti-4-Nb-4 (dark areas in SEM in Fig. 1c).

### 3.2 Small punch test

Dependence of force $F$ on punch displacement $v$ measured by small punch tests at the temperature of 600 °C is shown in Fig. 3. A notable yielding and higher plasticity of the Ti-0 alloy is obvious in comparison with the Ti-7 and Ti-4-Nb-4 alloys. The value of maximum recorded force can be used as an indicative parameter for the strength of material tested in small punch at constant deflection rate. The yield and ultimate tensile strength can be estimated using the correlations proposed by García et al. [7] or Purmenský and Matocha [8] listed in Table 2:

| Table 2. Empirical correlation formulas for determination of strength from SPT. |
|---------------------------------------------------------------|
| **Yield tensile strength** | **Ultimate tensile strength** | **Authors**          |
| $R_y^{SPT}$ | $R_m^{SPT}$ | $R_y^{SPT}$ = 0.346 \left( \frac{F_r}{h_0} \right)$ | $R_m^{SPT}$ = 0.277 \left( \frac{F_m}{u_m \cdot h_0} \right)$ |
| (MPa) | (MPa) | (1) | (2) | Garcia et al. [7] |
| $R_y^{SPT}$ = 1.67$F_y$ - 5.6 | $R_m^{SPT}$ = 0.35 \left( \frac{F_m}{u_m \cdot h_0} \right) + 23 | (3) | (4) | Purmenský and Matocha [8] |
where $F_m$ is the maximum force, $u_m$ is the deflection at $F_m$ and $h_0$ is the initial disc thickness. In this case of lower ductility alloys for simplification we consider the punch displacement equal to disc deflection. The value of $R_m^{SPT} = 154$ MPa is obtained for the alloy Ti-0 by Eq. 2. This is in good agreement with the $R_m = 165$ MPa obtained by a single mini tensile test at the same temperature [4]. The values of the ultimate tensile strength that can be obtained from the indicative data of Ti-7 and Ti-4-Nb-4 curves in Fig. 3 by the same correlation (Eq. 2) is equal to 979 MPa and 1088 MPa respectively. Table 3 shows a significant increase in the static strength of Ti-7 and Ti-4-Nb-4 compared to the Ti-0 alloy.

![SPT curve](image)

**Fig. 3. SPT curves of studied alloys at 600 °C.**

| Alloy       | $R_e^{SPT}$ (MPa) | $R_m^{SPT}$ (MPa) | Authors of formulas   |
|-------------|-------------------|-------------------|-----------------------|
| Ti-0        | 169               | 154               | García et al. [7]     |
| Ti-7        | 186               | 217               | Purmenský and Matocha [8] |
| Ti-4-Nb-4   | 463               | 1260              | García et al. [7]     |
| Ti-0        | 556               | 1397              | Purmenský and Matocha [8] |

### 3.3 Small punch creep test

The SPC tests were performed at 600 °C for all three alloys and also at 700 °C and 800 °C for Ti-7 and 800 °C for Ti-4-Nb-4 alloy. The time to rupture dependence on force is shown in Fig. 4a and the minimum deflection rate in Fig. 4b. The highest creep resistance has the Ti-4-Nb-4 followed by the Ti-7 followed by the Ti-0 alloy. The rate and rupture time comparison shows that the creep performance of the Ti-4-Nb-4 at 800 °C and Ti-7 alloy at 700 °C is similar as for Ti-0 alloy at 600 °C. Interesting is the comparison of the minimum deflection rates during a single test of Ti-7 alloy at 600 °C with stepped loading from 150 to 300 N (open squares in Fig. 4b) with the multiple tests with instant loading 225-300 N (filled squares in Fig. 4b). It suggests that instant high loading causes early cracking of the disc, possibly already in the loading phase. In contrary small load at the beginning of the test and small load increments prevent this early specimen cracking and result in the lower deflection rates. This effect is notable at load 250 N and higher. The SPC force and deflection rate can
be converted to equivalent stress and creep strain rate according to the methods described in [9-12] and compared with other creep resistant materials; this is however out of scope of this paper.

![Graph](image)

**Fig. 4.** SPC results a) time to rupture vs. force, b) minimum deflection rate vs. force.

### 3.4 Fractographic analysis

The fractographic analysis of ruptured SPC discs is shown in Fig. 5. The Ti-7 and Ti-4-Nb-4 alloys rupture at grain boundaries. Most notable it is for Ti-7 alloy in Figs. 5b and 5e; this specimen cracked even in the clamped area. The fracture of Ti-0 alloy is mixed due to large grains that are often larger than the specimen thickness 0.5 mm. The grain boundaries become well visible on the deformed sombrero-like specimen in Fig. 5d. The fracture surfaces in Figs. 5e and 5f correspond well with the microstructures.

![Images](image)
Fig. 5. SPC specimens after the tests at 600 °C: a) Ti-0, 125 N, b) Ti-7, 300 N, c) Ti-4-Nb-4, 400 N, d) Ti-0, 100N sombrero like specimen from interrupted test, e) Ti-7 fracture, f) Ti-4-Nb-4 fracture.

4 Conclusions
The small punch testing of three Fe-Al based alloys with and without Ti and Nb additions enabled to determine and compare their strength and creep behavior using specimens prepared from very small button ingots. The results show that a significant improvement in the mechanical properties can be reached by adding a small amount of alloying elements. A superstructure like nanocrystalline morphology was found in Ti-7 alloy, however the limiting factor for the mechanical properties is the strength of the grain boundaries and brittleness even at the experimental temperature of 600 °C. This seems to be improved for Ti-4-Nb-4 alloy despite the superstructure was not clearly visible in TEM.

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