# Laser-induced self-propagating reaction synthesis of Ti-Fe alloys

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Abstract Ti<sub>70.5</sub>Fe<sub>29.5</sub> alloy is synthesized using laserinduced self-propagating reaction synthesis (LSRS). The product mainly consists of  $\beta$ -Ti + TiFe eutectic. However, a given amount of oxygen-stabilized Ti<sub>2</sub>Fe phase is also found in the product due to high cooling rate and oxygen existence. The formation of the fine eutectic structure makes the alloy exhibit high hardness (9.34 GPa), high compressive strength (2609 MPa), and good relative compressibility (8.5%). The phase formation during LSRS can be divided into four stages: melting of Fe particle periphery, formation of a liquid-state Ti–Fe diffusion layer, eutectic reaction, and formation of oxygen-stabilized Ti<sub>2</sub>Fe phase.

# Introduction

High strength and ductile alloys are the objectives of many engineering alloys. Ti alloys in general suffer from low strength. One way to strengthen Ti alloys is to obtain metastable and even amorphous structures using rapid solidification techniques. It was reported that a  $Ti_{50}Ni_{25}Cu_{25}$  bulk glassy alloy had a very high tensile strength of 1800 MPa but was also extremely brittle [1]. The problem of high ductility was solved by introducing  $\beta$ -Ti dendrites into

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State Key Laboratory of Materials Modification, Dalian University of Technology, Dalian 116023, P.R. China e-mail: laser@dlut.edu.cn the Ti-based glassy alloys, obtaining a high compressive strength of 2.4 GPa and a good elasticity of 14.5% [2]. Recently, a binary Ti–Fe alloy prepared by a relatively slow cooling of about 10 K/s was found to possess a high mechanical strength exceeding 2000 MPa and good ductility of  $4 \sim 7\%$ [3].

Noticing that strong exothermic reaction due to large negative enthalpy of mixing ( $\Delta H_{\text{mix}} = -17$  Kj mol) can occur between Ti and Fe components in the Ti–Fe alloy systems, a new technique using Laser-induced Self-propagating Reaction Synthesis (LSRS) is proposed to fabricate the Ti–Fe eutectic alloys. In this article, the microstructure and properties of a LSRS Ti<sub>70.5</sub>Fe<sub>29.5</sub> alloy are investigated.

# **Experimental procedure**

Elemental powders of Ti (99.99% purity, -200 mesh) and Fe (99.90% purity, -200 mesh) were blended according to the eutectic composition of Ti<sub>70.5</sub>Fe<sub>29.5</sub>, and then the powdered mixture was pressed into a green compact of 6 mm in diameter and 5 mm in height. The ignition was carried out using CO<sub>2</sub> laser from the top of the green compact in an argon atmosphere. The laser ignition parameters were: laser power 0.8 kW, ignition time 6 s, and beam diameter 6 mm.

The microstructural characterization of the LSRS product was carried out using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The Vickers hardness in the vertical cross-section of the sample was measured using a load of 0.05 N. The compressive property was measured with MTS810 testing machine at a strain rate of 0.1 mm/min, the sample for the compressive testing was 4 mm in diameter and 4 mm in height.

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#### **Results and discussion**

# Microstructure of LSRS sample

Figure 1 shows an X-ray diffraction pattern of the LSRS product. It is seen that the product mainly consists of bodycentered cubic  $\beta$ -Ti, simple cubic FeTi, and face-centered cubic Ti<sub>4</sub>Fe<sub>2</sub>O phases, among which the Ti<sub>4</sub>Fe<sub>2</sub>O phase is generally regarded as oxygen-stabilized Ti<sub>2</sub>Fe intermetallics. The lattice parameter of  $\beta$ -Ti solid solution (0.3217 nm) is smaller than that of pure  $\beta$ -Ti (0.33065 nm) due to the dissolution of Fe, and the lattice parameters of the FeTi and Ti<sub>2</sub>Fe phases are found to be 0.2993 and 1.12978 nm, respectively.

Figure 2 shows a typical microstructure in the transverse cross–section of the product. The microstructure is featured with a fishbone-like  $\beta$ -Ti + TiFe eutectic. The interlamellar spacing of the eutectic is less than 0.6 µm. Since residual oxygen in the green compact is involved in the synthesis process, an oxygen-stabilized Ti<sub>2</sub>Fe phase with



Fig. 1 XRD pattern of the LSRS Ti<sub>70.5</sub>Fe<sub>29.5</sub> alloy



Fig. 2 Microstructure of the LSRS Ti<sub>70.5</sub>Fe<sub>29.5</sub> alloy



Fig. 3 The stress-stain curve of the LSRS Ti<sub>70.5</sub>Fe<sub>29.5</sub> alloy

an average size of 20  $\mu m$  is also found at the interface of the eutectic cell.

#### Properties

The average hardness of the product is 9.34 GPa. The strain-stress curve of the product under compressive testing is shown in Fig. 3. The product possesses a high compressive strength of 2609 MPa and a good relative compressibility of about 8.5%, of which the relative compressibility is given by  $h_0 - h_k /h_0 \times 100\%$ , where  $h_0$  and  $h_k$  are the height of the sample before and after compression, respectively. The fracture surface of the product shown in Fig. 4 manifests typical ductile (dimple) fracture is also found due to existence of the brittle Ti<sub>2</sub>Fe phase (as indicated by the arrow in Fig. 4).

The formation of the fine eutectic structure results in the high mechanical properties of  $Ti_{70.5}Fe_{29.5}$  alloy. The



Fig. 4 The fractography image of the LSRS Ti<sub>70.5</sub>Fe<sub>29.5</sub> alloy



Fig. 5 Microstructures of the LSRS product: (a) the originally compressed Ti and Fe particles locating in the unreacted zone; (b) the formation of Ti–Fe diffusion layer; (c) the linear distribution of Ti and Fe elements between Ti and Fe particles; (d) the formation of FeTi

compressive strength is comparable with that of Ti-based glassy alloys, while the ductility and hardness significantly exceed those of Ti-based glassy alloys.

# Process of microstructural evolution

The  $Ti_{70.5}Fe_{29.5}$  mixture is compressed in a copper die with conical cavity, and then ignited by laser from the bottom of the compact. Since the contact area per unit volume of the compact with the copper die is larger at the tip of the cone, the heat loss rate of the sample would increase when the reaction front approaches the tip of the cone. At the point where the combustion temperature becomes lower than the ignition temperature of the reaction, the reaction would be stopped. Since the initial, intermediate, and end reaction products are frozen in the quenched sample, the microstructural evolution can be observed and analyzed.

phase at the peripheries of Fe particles; (e) the nucleation and growth of  $\beta$ -Ti + FeTi eutectic; (f) the formation of oxygen-stabilized Ti<sub>2</sub>Fe phase between the eutectic cells

As shown in Fig. 5a, the unreacted zone located at the tip of the quenched sample consists of originally compressed Ti and Fe particles. Once the combustion wave front approaches the reacting zone, the peripheries of Fe particles are melted first. Then, the melting spreads over the entire Fe particles, and finally welds the Ti and Fe particles. Since diffusion of Fe into Ti particles is followed by a decrease of melting point, a liquid-state Ti-Fe diffusion layer between Ti and Fe particles (gray layer in the Fig. 5b) is formed. The composition distribution of the diffusion layer revealed by EDS is shown in Fig. 5c. With the further proceeding of the diffusion, the TiFe phase is formed at the peripheries of Fe particles, and is followed by a eutectic reaction where the Ti-enriched melt forms  $\beta$ -Ti and TiFe (Fig 5d, e). Finally, Fe particles are completly dissolved. Between the eutectic cells are presumably oxygen-stabilized Ti<sub>2</sub>Fe grains (Fig. 5f).

#### Conclusions

A laser-induced self-propagating reaction synthesis method has been successfully applied in the fabrication of Ti–Fe alloy. The LSRS of the alloy shows that the product mainly consists of FeTi +  $\beta$ -Ti eutectic. However, a given amount of oxygen-stabilized Ti<sub>2</sub>Fe phase is also found in the product due to high cooling rate and oxygen existence. The alloy exhibits high hardness, strength, and plasticity due to the formation of the fine eutectic structure. The formation process of the product during self-propagating reaction synthesis can be divided into four stages: melting of Fe particle periphery, formation of Ti–Fe diffusion layer, eutectic reaction, and formation of oxygen-stabilized  $Ti_2Fe$  phase.

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