Development of Rolling Technology for an Iron-based Shape-Memory-Alloy

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Abstract. Low cost Fe-Mn-Si based shape memory alloys (SMA) have drawn much attention during the last two decades as a cost-effective alternative to the expensive Ni-Ti based SMA. In particular, the alloy Fe-17Mn-5Si-10Cr-4Ni-1(V,C) (mass%), which has been developed at Empa shows very promising properties with regard to potential commercial applications in civil and mechanical engineering. This alloy has a higher reverse transformation temperature and larger thermal hysteresis in comparison to the Ni-Ti based alloys, which is adequate for producing stable recovery stresses at room temperature. Furthermore, recovery stresses of up to 300 MPa after heating to only 160 °C can be achieved without so-called ‘training’ treatment. Furthermore, the alloy can be easily and cost effectively produced under standard air melting and casting conditions. For availability of these heavily microstructure dependent skills for civil and mechanical engineering, e.g. as prestressing elements in concrete structures or coupling/clamping devices, a process chain for manufacturing is necessary. Therefore, a hot and cold rolling technology for strip production with thermal heat treatment processes was developed at TU Bergakademie on base of experimental simulation results. The last one helps to understand the dependencies of deformation parameters, the deformation behavior and their influence to the microstructure evolution in correlation to the recovery.

This paper discusses the basic material properties, recovery stress formation behavior and finally the feasibility of the alloy as reinforcing elements in civil engineering applications by using a rolling technology for flat products.

Introduction

Shape memory alloy (SMA) steels based on the Fe-Mn-Si alloy system are promising candidates for a variety of advanced civil engineering applications, since they have a wide transformation hysteresis, high elastic stiffness and strength and are relatively inexpensive to produce [1]. These advantages make them promising as a cost-effective alternative to conventional NiTi-based SMAs for applications requiring high shape memory stresses, e.g. constrained recovery applications such as external confinement for reinforced concrete columns [2] or as prestressing reinforcement in prestressed concrete [3;4].

The shape memory effect in Fe-Mn-Si alloys is a result of a mechanically induced phase transformation from austenite (face centered cubic phase, γ) to martensite (hexagonal close packed phase, ϵ) and its reversion upon heating [5]. Under pure mechanical loading, stress induced martensite forms and the resulting strain can exceed the elastic strain limit of the alloy. This strain can be recovered by the reverse transformation due to the same crystallographic path of the two transformations [6].

Recently, some of the present authors reported that Fe-SMAs with finely dispersed VC particles have very promising properties for commercial civil engineering applications, in particular a very high recovery stress after heating to only 160 °C, high fatigue strength as well as a high resistance against corrosion [7-10]. In a feasibility study, the Fe-SMA strips (Figure 1) were used to prestress concrete bars by having been centrally embedded [11]. The Fe-SMA strips were activated by resistance heating. Compressive stresses in the range of 3 MPa in the concrete section were obtained. Furthermore, it was shown in [12], that the Fe-SMA strips could be used for structural strengthening of reinforced concrete (RC) beams. The shape memory effect of the Fe-SMAs was used to prestress concrete beams. The strips were embedded in grooves at the bottom of the RC beam, similar as the near surface mounted (NSMR) strengthening technique with FRP laminates. Three beams prestressed by Fe-SMA strips were compared with one beam strengthened by non-activated Fe-SMA strips. The observed higher cracking load and the smaller deflections for a given service load proved that strengthening of RC beams with prestressed Fe-SMA strips worked well [13].

These studies confirmed the high potential and cost efficiency of these Fe-SMAs for advanced civil engineering applications.

Fig. 1: Final Fe-SMA strips with and without ribs.

The shape memory properties of Fe-Mn-Si based SMAs are strongly dependent on their microstructure. For the availability of SMA products with reproducible properties, a process chain for manufacturing is necessary. Therefore, a hot and cold rolling technology for the strip production with thermal heat treatment processes was developed on base of experimental simulation results.

Material and Experimental Simulation

The chemical composition of the alloy was Fe-17Mn-5Si-10Cr-4Ni-1(V,C) (mass%). An approximately 95-kg alloy ingot was induction melted under normal atmospheric conditions and cast into square mould. The ingot was cast with feeder head and exothermal anti-piping powders to prevent cavities.

A very important material parameter for development and optimization of metal forming processes is the flow stress or the flow curve dependent on strain rate, deformation temperature and true strain. This material-specific parameter is necessary to predict the required amount of force and work for each deformation step. With help of these knowledge and realisation is it possible to lay out the suitable deformation tools as well as the forming plants, but also for the numerical simulation is the information about the flow behaviour of the materials indispensable.

The evaluation of the flow curves was carried out by twofold occupied cylindrical compression tests under process-oriented conditions at the multifunctional deformation simulator Gleeble HDS-V40 at the Institute of Metal Forming. Isothermal flow curves (Figure 2) result from the recorded force-displacement data considering the correction of the measured values with regard to dissipation energy and friction. These flow curves are base for the calculation of the rolling forces, which are limited in the semi-continuous rolling mill at the Institute of Metal Forming [14].

Prior to deformation, the samples were conductively heated up with 5 K/s to 1150 °C. Then, the temperature was kept constantly for one minute followed by a cooling process to the final forming temperature of 950 to 1150 °C. After that, the samples were compressed continuously to an effective strain of $\varphi_v = 1$ using several deformation rates (1 up to 10 s⁻¹).

Fig. 2: Flow curves for different strain rates and temperatures (left: 950 °C; right: 1100 °C)

Multi-step deformation tests of cylindrical samples were performed in the Gleeble HDS-V40 (see Figure 3) considering microstructural evolution as well as for the determination of the technological process window for rolling tests.

Fig. 3: Time-temperature-regime for multi-step deformations ($\varphi_{\text{total}} = 1.2$) in Gleeble HDS-V40

The tests were performed based on the plant-specific characteristics of the rolling mill. Two different forming strategies are summarised in Table 1.

Table 1: Deformation strategy for multi-steps for forecast of microstructure evolution

	Deformation strategy 1	Deformation strategy 2
Homogenization in °C	1150	
True strain distribution (3 deformation steps)	0.45 + 0.4 + 0.35	0.5 + 0.45 + 0.25
Pause time between each deformation steps in s	10	
Strain rate for each deformation in 1/s	1	10

Furthermore, these experiments aimed at the identification of grain size evolution, precipitates and softening behaviour as functions of temperature, true strain, pause time and strain rate (cf. Figure 4).

Fig. 4: Comparison of influence of different deformation strategies at microstructure evolution (left: strategy 1; right: strategy 2)

By comparing both deformation routes with additional experiments at different temperature-true strain combinations) it can be concluded that the final deformation temperature in combination with the strain rate have primary influence on the microstructure evolution and ultimately on the mechanical properties. The identified minimum deformation temperature was approximately 1000 °C for obtaining small grains and fine precipitates in the matrix. However, when a too high starting temperature is selected, the dissipation energy during the deformation can lead to damage by grain boundary melting. The microstructure obtained using strategy 1 shows semi recrystallized grains and the average measured grain diameter was approximately 5 µm. In comparison, the recrystallized grain diameters obtained using strategy 2 were on average smaller than 4 µm, and the carbide precipitates were smaller and more homogeneously distributed over the whole steel matrix. The aim of the subsequent rolling test for the production of strips was to disperse the VC precipitates in a fine and homogenous manner (cf. Figure 4, right), and not on the grain boundaries (cf. Figure 4, left). These overall results of the experimental simulation were used for the process design and the validation with rolling experiments under laboratory conditions.

Rolling Technology for Flat Products

The raw material for all test at the Institute of Metal Forming was a cast ingot with a weight of 95 kg and a cross-section of (190...140) mm x (190...140) mm x 440 mm (w x h x l). Hence, an open die forging technology was selected considering the results of the experimental simulation. The forging was realised on the 1000 t oil-hydraulic universal forging press in several heats (starting and reheating temperature of 1150 °C for 30 min after reaching the temperature in the core; before the first forming step as measured with a thermocouple) for minimising the risk of edge cracks and at the beginning with true strains of $\varphi \leq 0.3$ to transfer the casting microstructure with dendrites gradually into a globular deformation microstructure. The final forging cross-section after the third reheating procedure was 85 mm x 85 mm followed by the hot strip rolling process on a 2400 kN duo rolling mill. A hot rolled strip with a thickness of 1.7 - 1.9 mm could be produced with three reheating operations and 15 reversing rolling steps. The complete manufacturing process is shown in Figure 5.

Fig. 5: Hot deformation strategy for SMA from cast ingot to 1.7-1.9 mm strip

Before the cold-rolling process for imprinting the ribs, the SMA strips were solution heat treated at 1100 °C for 30 min. After that, a precipitation heat treatment at 850 °C for 2 h was performed to form fine VC particles in the SMA matrix.

Material Properties of Fe-SMA

Figure 6 shows across-sectional optical micrograph of the strip material. Rather equiaxed grains with diameters between 10 and 40 μm can be observed

Fig. 6: Microstructure of SMA strip material after hot rolling and heat treatment.

Figure 7 shows the stress-strain behavior of the Fe-SMA strips during prestraining, necessary to produce martensite phases. It is visible that they start to develop at tensile stresses of approximately 300 MPa and strains of approximately 0.25 % (inclination decreases). Tensile strains at failure of the Fe-SMA strips are approximately 10 % with a tensile stress of approximately 700 MPa [11]. Then, the applied tensile force was released to 10 N and the remaining strain was measured. It can be observed that the behavior during unloading deviates from Hook's law since the stress-strain curve during unloading does not follow a linear course. This curved progression indicates the so-called pseudo-elastic effect and has been reported already in [8,10]. When the stress is released, a part of the martensite is not stable anymore at room temperature and transforms spontaneously back into austenite.

Fig. 7: Stress-strain curve of two Fe-SMA strip test samples during prestraining to produce martensite phases.

The recovery stresses determined in a tensile testing machine with a climate chamber after the prestraining were approximately 250 – 300 MPa [11]. Two such experiments are shown in Figure 8. It is visible that the preload reduces at the beginning of the test because of temperature expansion, then, at approximately 40 °C, the phase transformation starts and the stress increases up to the maximum heating temperature of 160 °C. During cooling, stress continues to increase because of heat shrinkage.

Fig. 8: Stress-temperature curve of two Fe-SMA strip test samples (after prestraining) to determine the recovery stress.

Civil Engineering Applications

The company re-fer AG (www.re-fer.eu) was founded in 2012 based on the developments at Empa with the aim of producing and selling the patented, iron-based SMAs for civil engineering applications. In addition to the patented alloy, re-fer AG and Empa hold several patents for specific civil and mechanical engineering related applications using Fe-SMA bars and strips. Fe-SMA reinforcements can be used for strengthening existing structures and also for new structures. The main applications which are envisaged at the moment are:

- ribbed Fe-SMA bars for reinforcing shotcrete for strengthening existing reinforced concrete structures. Such applications could for instance be performed on the bottom side of a concrete slab
- ribbed Fe-SMA for prestressed near surface mounted reinforcement for strengthening existing reinforced concrete structures. Possible applications could be negative bending moments on the top side in concrete cantilever elements or over column supports
- ribbed Fe-SMA bars for precast concrete element production
- ribbed Fe-SMA bars for prestressed shear strengthening in existing or new concrete constructions.
- end-anchored Fe-SMA plates for bending strengthening

Conclusions and Outlook

Ribbed Fe-SMA strips with large-scale dimensions for structural strengthening in civil structures were manufactured. The identified rolling parameters must transfer to an industrial rolling production line. Furthermore, a continuous rolling technology for manufacturing of long products must be adapted from the existing level of knowledge.

The recovery stress (i.e., prestress after activation) of the Fe-SMA strips are in the range of 250 – 300 MPa. Several feasibility studies with experimental investigations have shown their performance in prestressing reinforced concrete structures.

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