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Mechanical properties of a new Fe-Ni-based Invar alloy doped with Ti, V and C

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Abstract: The Fe-36 wt.% Ni binary Invar alloy has low mechanical strength, with an ultimate tensile strength (σ_{UTS}) of less than 500 MPa. This greatly limits its practical applications. An increase in mechanical properties was achieved by the development of a new Invar alloy, Fe-32 wt.% Ni-2.7 wt.% (Ti, V, C). Alloying the binary alloy with carbon, titanium and vanadium, followed by deformation and heat treatment, allows increasing the ultimate tensile strength to $\sigma_{UTS} = 1185 \pm 3$ MPa, which is more than twice that of classical Invar. At the same time, it retains acceptable ductility ($\delta = 4.6 \pm 0.3\%$) and a low coefficient of thermal expansion at room temperature (about 1.2×10^{-6} K^{-1}). The high strength of the new Invar alloy can be explained by the fact that, as a result of alloying followed by deformation and heat treatment, several hardening mechanisms are implemented, namely, dispersion, solid-solution, deformation and grain-boundary ones. However, the study showed that the potential for dispersion strengthening is not realized to its fullest extent due to carbide coarsening during hot forging and incomplete dissolution during further annealing. Further work will focus on optimizing the hot working and heat treatment parameters to prevent carbide growth and increase the strength of the alloy. These results will enable the use of the new Invar alloy in advanced industrial sectors where high accuracy, dimensional stability and the ability to withstand significant mechanical loads are required.

Keywords: high-strength Invar alloy, low coefficient of thermal expansion, quenching, aging, microstructure, mechanical properties

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1. Introduction

The binary Invar alloy, Fe-36 wt.% Ni (hereafter referred to as Fe-36Ni), is commonly used in components for devices requiring stable dimensions, precision instruments and microelectronics. However, its low mechanical properties (typically $\sigma_{UTS} < 500$ MPa) restrict its use in heavily loaded and large-scale structures. In order to expand the potential applications of the Invar alloy, its strength properties must be significantly improved. In this case, Invar alloy would be of interest for the production of metal structures for membrane tanks used for transporting liquefied natural gas (LNG) [1]. The Invar membrane layer is assembled from 0.7 mm thick sheets. The layers are welded together to ensure the structure is airtight. Due to the unique property of the Invar to expand/contract minimally when the temperature changes ensures that the sealed membrane layer doesn›t crack during the LNG tank filling process (where the gas temperature is about $T = -163$°C). Heating the gas during transportation does not change the geometric dimensions of the tank wall. Invar alloy is also used to manufacture high-temperature power transmission wires. For example, they are used to increase the strength of (Z)TACSR/HACIN wires and reduce sagging [2]. Using heat-resistant aluminium for the current-carrying part of the wire increases throughput, and using a super-heat-resistant welded alloy enhances this effect further. Another modern application of Invar alloy is its use as a mould material in the production of composite black carbon fiber wings, as seen in the new medium-range MC-21 aeroplane [3]. In all these cases, using an Invar alloy, which has a higher strength than the Fe-36Ni alloy, will improve the cost-effectiveness. High-strength Invar alloys will undoubtedly have many other applications in the future. Therefore, developing an Invar alloy that combines high strength with a low coefficient of thermal expansion (CTE) is a pressing issue.

Invar alloys have been the focus of research for quite a long time (over a century). Initially, the physical properties of binary Fe-Ni alloys were studied, with little attention was given to its strength. The Fe-36Ni alloy was mainly associated with practical applications. At a certain point, it was realized that the low strength of the alloy was an obstacle to its wider use. In [4], the potential for strengthening the Fe-36Ni alloy through refining its microstructure to the submicron level was explored. The alloy was subjected to equal-channel angular pressing, which significantly reduced its grain size. The average grain size decreased from 100 μm to 180 nm. This led to an increase in ultimate tensile strength from 490 to 912 MPa. At the same time, the ductility of material increased (from $\delta = 40\%$ to $\delta = 52\%$). An even higher ultimate tensile strength of 1420 MPa was achieved in [5], where the grain size of approximately 100 nm was obtained using high pressure torsion. Reducing the grain size to the submicron level has a strong effect on grain boundary strengthening. Unfortunately, this is not feasible on an industrial scale.

Various attempts to strengthen the Fe-36Ni alloy by adding elements that provide dispersion and solid-solution strengthening have resulted in a significant increase in CTE. For example, adding 0.5 wt.% of Be to an alloy increases its ultimate tensile strength to 1100 MPa due to the formation of Ni_3Be intermetallic particles. However, this simultaneously results in a significant increase in the CTE and an unacceptable decrease in ductility [6]. A similar effect is observed when strengthening particles of the Ni_3Al intermetallic compound are introduced [7].

Recent studies have shown that introducing C in small quantities (up to 0.7 wt.%), as well as carbide-forming elements such as Mo (2.3 – 3.4 wt.%) and V (1–1.5 wt.%), can significantly improve strength properties while maintaining low CTE (maximum σ_{UTS} =1100 MPa and CTE $< 2\times10^{-6}$ K^{-1}), although ductility decreases noticeably ($\delta \approx 5\%$) [8 –12]. These studies showed that adding up to 0.6 wt.% of C to Fe-Ni alloys decreases their CTE and strengthens them. The greatest decrease in CTE is observed at a Ni content of 32 – 34 wt.%. Moreover, adding carbon to Fe-Ni alloys near the Invar composition increases the temperature range over which a low CTE is maintained and lowers the onset temperature of martensitic transformation. In [8], it is recommended to add up to 0.7 wt.% C, assuming that some of this (up to 0.3 wt.%) forms V carbide and the remainder (up to 0.4 wt.%) remains in solution. In [12], it was found that, when Invar alloys were heated to a quenching temperature of T=1200°C, a significant volume fraction of carbides was retained (rather than dissolved) in their microstructure. The highest volume fraction of carbides is found in the Invar alloy alloyed with both Mo and V. These carbides inhibit grain growth during the quenching process and help to preserve the fine-grained microstructure achieved through previous deformation. This is important because subsequent ageing enables carbides to precipitate uniformly primarily along grain boundaries. In [9], it was established that the quenching temperature was of significant importance during the heat treatment of Invar alloys alloyed with C, V, and Mo. Quenching at 1250°C followed by ageing at 650°C increases the strength by 300 – 400 MPa more than quenching at 1200°C followed by ageing at 650°C. During the development of new Invar alloys, Ti, which is the strongest carbide-forming element, was not used as an alloying element in these studies. In addition, little attention has been paid to forming a homogeneous, fine-grained microstructure in Invar alloys during processing. This may be a key to achieve a uniform distribution of dispersed carbides, and ultimately to improve the strength and ductility of these materials. One method of forming such a microstructure is multiple isothermal forging [13 –16].

Significant efforts to improve the strength of Invar alloys have been made by Chinese researchers [17– 20]. In [17], the Fe-36.7Ni-4.41Mo-0.26C (wt.%) alloy was investigated. This alloy was subjected to homogenizing annealing at 1250°C, hot rolling to form a rod at the same temperature, quenching at 1050°C, and ageing at 525°C. In the work, the ultimate tensile strength of σ_{UTS} = 820 MPa and high relative elongation $\delta = 35\%$ were obtained. At the same time, it was possible to maintain a low CTE of 3.37×10^{-6} K^{-1}. A uniform distribution of Mo_2C particles was achieved in the austenitic matrix. One disadvantage of the alloy was the rapid aggregation and growth of Mo carbide particles during ageing. Another Fe-36.7Ni-4.08Mo-0.11V-0.11Ti-0.006N-0.29C (wt.%) alloy was subjected to homogenizing annealing at 1250°C, hot rolling to form a rod at the same temperature, quenching at 1250°C, and ageing at 780°C [18]. Such treatment resulted in an ultimate tensile strength of σ_{UTS} = 940 MPa and a high relative elongation of $\delta = 35\%$. At the same time, it was possible to maintain a low CTE of 2.33×10^{-6} K^{-1}. The increased strength was due to the precipitation of Ti(C, N) and VC nanoparticles during the initial and middle stages of ageing. However, the rapid aggregation and growth of Mo carbide particles at the final stage of ageing unexpectedly resulted in a decrease in strength and an increase in the CTE value.

The work [19] investigated the Fe-36.6Ni-1.5Cr-1.3Mo-1.1V-0.22C (wt.%) alloy, which was hot rolled onto a plate at T=1150°C. The plate was then cut into rods, which were warm rolled at T= 750°C, quenched at T=1250°C (after 3 hours holding), and aged at T= 650°C. As a result of this treatment, the alloy demonstrated an ultimate tensile strength of σ_{UTS} = 921 MPa, a relative elongation of δ =11.2%, and a low CTE of 3.2×10^{-6} K^{-1}. The study showed that warm rolling promotes the formation of a large number of twin boundaries, ensuring uniform precipitation of dispersed V_8C_7 particles. In this case, Mo carbide does not form at all. In [20], the possibility of combining strain and dispersion hardening was investigated on the Fe-37.5Ni-5.5Cr-2.0Mo-0.1Ti-0.1V-0.02N-0.25C (wt.%) alloy. This alloy was hot forged to create a blank for rolling. The blank was then hot rolled, quenched at T=1250°C (after 4 hours of holding), and cold drawn, resulting in an 88.9% decrease in cross-sectional area. As a result, the ultimate tensile strength increased to 1166 MPa. At the same time, ductility decreased significantly (δ <1%) and the CTE increased to 4.85×10^{-6} K^{-1}. The increase in tensile strength is explained by strain hardening, grain boundary strengthening and dispersion strengthening caused by the formation of VC and Mo_2C nanoparticles during deformation.

Thus, literature data suggests that Fe-Ni-based Invar alloys can be significantly strengthened by alloying with carbon and carbide-forming elements while maintaining low CTE. To maximize the strength of Invar alloys, all known strengthening methods must be implemented, i. e., dispersion, solid-solution, deformation, and even grain-boundary strengthening ones. This requires further optimization of the composition and processing conditions. In particular, the alloying potential of titanium, the strongest carbide-forming element, remains unclear. The lack of sufficient carbon in the alloys under study was a disadvantage of all the above-mentioned studies. This did not allow for the full implementation of both dispersion and solid-solution strengthening.

This article examines the mechanical properties and microstructure of a new Invar alloy, Fe-32Ni-2.7(Ti, V, C) (wt.%), that has been deformed and heat-treated. The chemical composition and modes of deformation and heat treatment were chosen based on a literature review.

2. Materials and experimental

Invar alloy ingots measuring ∅45 mm × 16 mm were produced using the argon-arc melting process in a laboratory melting unit. High-purity components were used in the melting process, which was carried out in a high-purity argon atmosphere. To achieve high chemical homogeneity, each ingot was remelted multiple times. Energy dispersive analysis confirmed that the resulting alloy composition was close to the nominal ingot composition: Fe-32Ni-2.7(Ti, V, C) (wt.%). After melting, the ingots were weighed using an analytical balance, which also served as an indirect method of monitoring the chemical composition of the alloys.

After melting, the ingots underwent homogenizing annealing at T=1250°C in a vacuum furnace for τ = 2 hours. A homogeneous fine-grained structure was obtained through hot forging. This was carried out in four passes under quasi-isothermal conditions, with a 90° deformation axis change. The forging was done on a 100-ton EU-100 hydraulic press. The press was equipped with an isothermal die block made of a heat-resistant nickel alloy. The ingots were heated in the furnace to T=1050°C and then quickly transferred to an isothermal die block heated to T= 950°C. They were then deformed by compression in three stages with a strain of ε = 50% in each stage and ε = 70% in the fourth stage. After forging, the parallelepiped-shaped blanks with dimensions of 30×40×13.5 mm^3 were placed in a stainless steel shell. The resulting package was heated to T= 900°C and rolled on a DUO-300 rolling mill in several passes with intermediate heating to a relative reduction of approximately 50%. The rolled blanks were heated to T=1250°C, held for τ=15 min, and quenched in water. After quenching, the blanks were removed from the shell and then cold rolled to a relative reduction of about 70%. Figure 1 shows a schematic diagram of processing the Fe-32Ni-2.7(Ti, V, C) (wt.%) alloy.

Flat specimens for mechanical testing were cut from rolled alloy blanks and then ground on abrasive paper with a grit number of at least P2500. Tensile tests were conducted at room temperature on three specimens with working area dimensions of 15×3×1.6 mm^3. Microhardness (HV_{1000}) was measured using an ITV-1-AM microhardness tester. Microstructural studies were performed using a Mira-3 LMH scanning electron microscope (Tescan) in backscattered electron (BSE) mode. Surface preparation of the samples for microstructural studies was carried out in two stages: (1) mechanical grinding, followed by polishing on velvet paper; (2) electrolytic etching in a 1:9 volume ratio solution of $HClO_4$ and $C_4H_{10}O$ at a constant voltage of 30 V for 30 seconds. Energy-dispersive (EDS) analysis was used to evaluate the chemical composition of the matrix and the carbides within it. The volume fraction of carbides was estimated using the "Particles" program built into the scanning electron microscope for digital image contrast analysis. The source was a microstructure image in compositional contrast mode. This method yields the proportion of pixels in a digital image of the microstructure that are closer to the black in grayscale terms. This indicates that the average atomic number of these carbide phases is lower than that of the matrix. CTE measurements were performed in the temperature range from room temperature to 200°C using a dilatometer with a displacement sensor based on a differential transformer.

3. Results

3.1. Microstructure

Figure 2a shows the as-cast microstructure of Fe-32Ni-2.7(Ti, V, C) alloy. It is characterized by a dendritic structure with dispersed carbide precipitations. The grain size is several hundred microns and the carbide size in between 1 and 5 μm. Some carbides are located along the

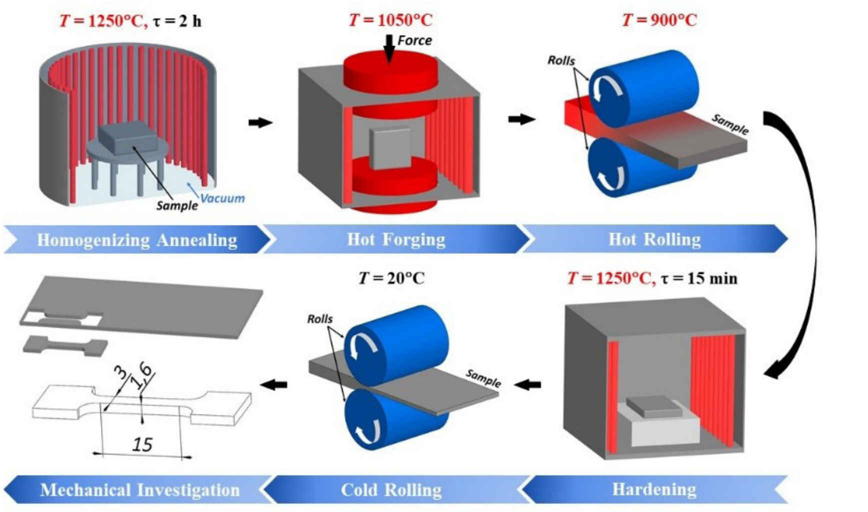

Fig. 1. (Color online) Processing scheme for the Fe-32Ni-2.7(Ti, V, C) (wt.%) Invar alloy. Sample sizes are given in mm.

grain boundaries and can be about 10 μm long (see Fig. 2b). The volume fraction of carbides is γ = 3.13% (see Table 1). Following homogenizing annealing and subsequent furnace cooling, the grain size remains unchanged and the carbide size does not increase (see Fig. 2c). Conversely, due to ageing during cooling, more dispersed carbides measuring less than 1 μm in size are formed (Fig. 2d). The volume fraction of carbides remains at γ = 3.11%. Hot forging significantly refines the microstructure (Fig. 2e), forming a uniform fine-grained microstructure with a typical grain size of d = 5–10 μm. During this process, some carbides noticeably enlarge, reaching sizes of 10 μm or more. However, their volume fraction does not change significantly (γ = 3.16%).

During subsequent hot rolling at 900°C, elongation of the grains in the rolling direction occurs, as well as a noticeable increase in the volume fraction of carbides up to γ = 5.15%, apparently due to dynamic ageing (see Fig. 2f). In this case, several large carbides measuring about 10 μm are also observed. After hardening at T = 1250°C for τ = 15 min and water quenching, the grain size increases slightly to d = 15–40 μm. Numerous annealing twins are formed and the volume fraction of carbides decreases significantly to γ = 3.73% (see Fig. 2g) due to the dissolution of relatively small carbides. At the same time, carbides with a size range of 5–10 μm are preserved; in other words, large particles do not dissolve. As a result of cold rolling, the microstructure consists of macroscopic bands, predominantly oriented at an angle of 45° to the rolling direction. During cold rolling, dynamic ageing occurs, leading to a noticeable increase in the volume fraction of carbides to γ = 4.68%. The sizes of these carbides range from nanometres to 20 μm (see Fig. 2h).

3.2. Chemical composition

Figure 3 shows the microstructure and elemental maps of the same sample region of the alloy after hot forging. Qualitatively, the particles are enriched in Ti, V and C, and a relatively uniform distribution of these elements is observed in the matrix. Tables 2 and 3 show the typical chemical composition of the carbides and matrix after different processing stages. The carbides have a complex chemical composition, containing predominantly Ti (the strongest carbide-forming element), as well as smaller amounts of V, Fe and Ni. EDS analysis of the chemical composition of the new Invar alloy showed that it presumably contained Ti_2VC ternary carbide. Table 3 also shows the typical matrix composition. As expected, Ti is primarily present in the carbides, while V remains largely in the solid solution.

Table 1. Volume fraction of carbides following different types of processing.

Condition, processing	Cast condition	Homogenizing annealing 1250°C, τ = 2 h	Hot forging 1050°C	Hot rolling 900°C	Annealing, quenching 1250°C, τ = 15 min	Cold rolling 20°C
Carbide size, γ (%)	3.11	3.13	3.16	5.15	3.73	4.68

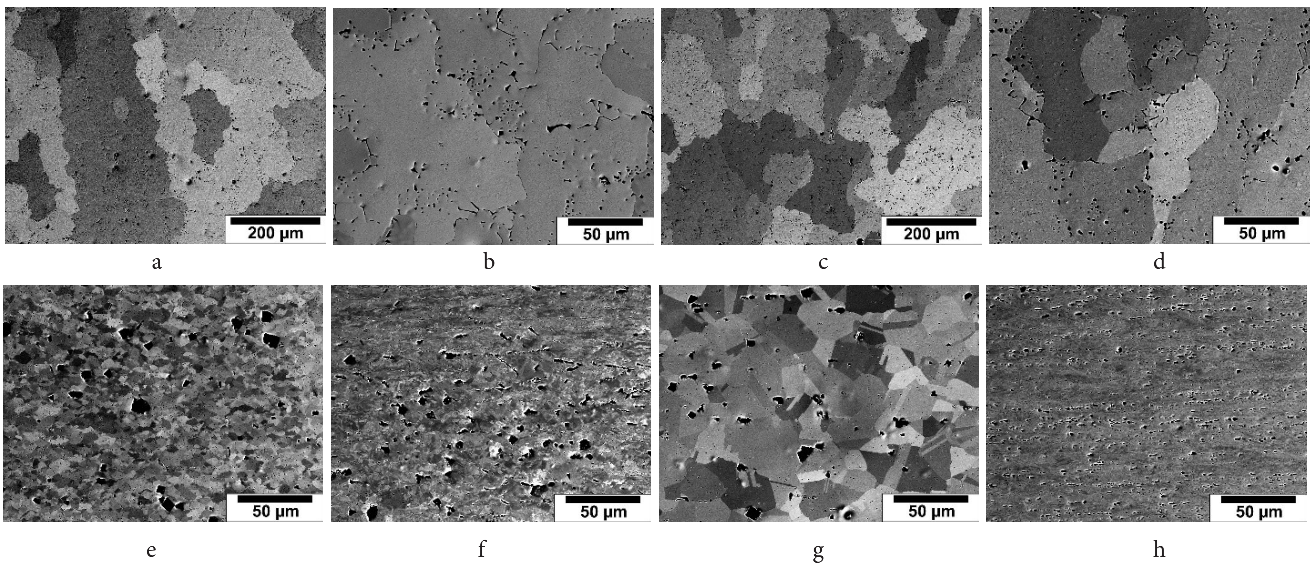

Fig. 2. Microstructure of the sample: in the as-cast condition (a, b); after homogenizing annealing at 1250°C for 2 hours (c, d); after hot forging at 1050°C (e); after hot rolling at 900°C (f); after hardening at 1250°C for 15 min and quenching in water (g); after cold rolling (h).

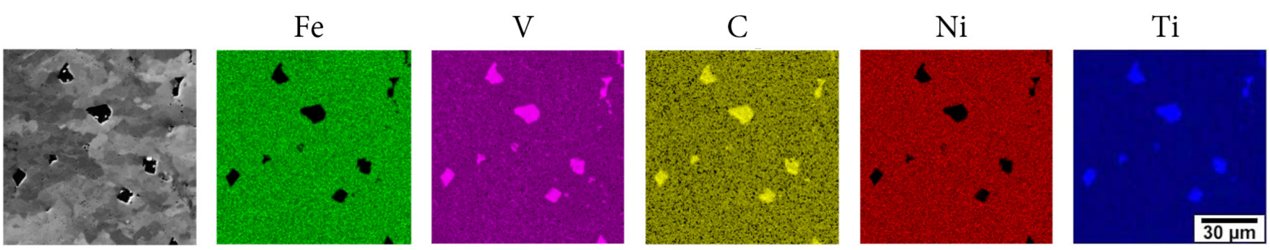

Fig. 3. (Color online) Microstructure of the alloy after hot forging and a map of the distribution of chemical elements in carbides.

3.3. Mechanical properties

As has been discussed above, dynamic ageing occurs during cold rolling after annealing at 1250°C for 15 minutes and water quenching. This leads to the precipitation of a significant number of carbides. This raises the question: can carbides also precipitate during static ageing? To answer this, we plotted the dependence of microhardness on aging temperature $HV_{1000}(T)$ (Fig. 4), which shows that static aging does not occur. Mechanical tensile tests of Invar after cold rolling showed an ultimate tensile strength of σ_{UTS}=1185±3 MPa, and an elongation at failure of δ=4.6±0.3%.

3.4. Thermal expansion

CTE measurements were performed on the new Fe-32Ni-2.7(Ti, V, C) Invar alloy in its cold-rolled condition. Figure 5 shows the curves of the CTE as a function of temperature for the new Invar alloy Fe-32Ni-2.7(Ti, V, C) (blue curve) and the binary alloy Fe-36Ni (red curve). At room temperature, it can be seen that the CTE for both alloys is close to $\alpha=1.2\times10^{-6}$ K^{-1}. As the temperature increases, α begins to grow monotonically for both alloys. In the temperature range of 25–140°C, the CTE of both alloys is practically identical. However, for the new alloy, growth occurs faster than for the binary alloy starting from a temperature of about 140°C. This difference between the curves is due to the fact that, when heating above 140°C, the CTE of precipitated carbide nanoparticles contributes significantly to the increase in CTE of the new alloy. This leads to more intense growth of α.

4. Discussion

The research has revealed several unexpected findings. Firstly, the additional alloying of the Invar alloy with Ti, as opposed to the main alloying elements of V and V+Mo in [8–12], leads to a more than twofold increase in the volume fraction of carbides. This is apparently due to the role of Ti as a stronger carbide-forming element than V and Mo. However, an increase in the volume fraction of carbides does not significantly affect the CTE compared to a binary alloy (approximately $\alpha=1.2\times10^{-6}$ K^{-1}). Secondly, hot forging at T=1050°C causes strong enlargement of individual carbides (up to 10 μm or

Table 2. Elemental composition of carbides (wt.%).

Condition / processing	Fe	Ni	Ti	V
As-cast condition	1.30	0.21	80.80	6.75
Homogenizing annealing 1250°C, τ=2 h	1.94	0.75	65.13	16.32
Hot forging 1050°C	1.96	0.76	67.93	14.12
Hot rolling 900°C	3.80	0.50	63.33	16.88
Annealing, quenching 1250°C, τ=15 min	1.97	0.84	58.47	14.64
Cold rolling 20°C	1.76	0.28	65.25	14.64

Table 3. Elemental composition of matrix (wt.%).

Condition / processing	Fe	Ni	Ti	V
As-cast condition	66.36	32.70	0.21	0.73
Homogenizing annealing 1250°C, τ=2 h	66.48	32.90	0.07	0.55
Hot forging 1050°C	66.64	32.80	0.10	0.46
Hot rolling 900°C	66.58	32.97	0.09	0.36
Annealing, quenching 1250°C, τ=15 min	66.47	32.86	0.12	0.56
Cold rolling 20°C	66.68	32.68	0.09	0.55

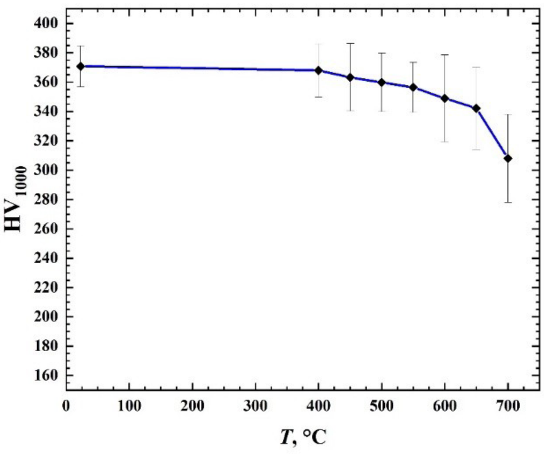

Fig. 4. Microhardness (HV_{1000}) as a function of ageing temperature (ageing time: τ=1 hour).

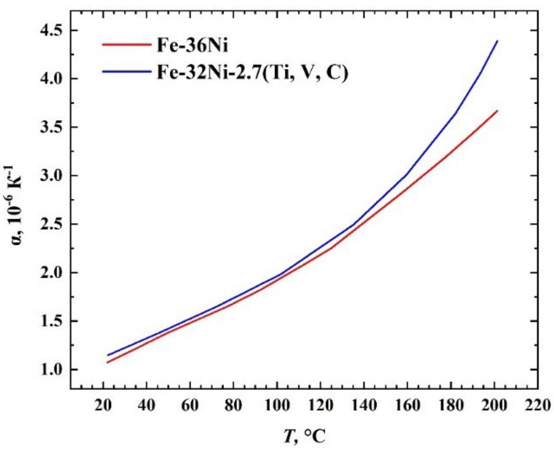

Fig. 5. (Color online) CTE as a function of temperature for the new Invar alloy Fe-32Ni-2.7(Ti, V, C) (blue curve) and binary alloy Fe-36Ni (red curve).

more). The growth of carbides during hot forging, when recrystallisation takes place and diffusion is activated, is not surprising in itself. However, their strong enlargement in comparison with recrystallized grains is surprising. Hot forging at T=1050°C appears to contribute to this to a large extent. Thirdly, alloying with two of the strongest carbide-forming elements significantly complicates the dissolution of the formed complex carbides during subsequent annealing at T=1250°C and a significant volume fraction of carbides remains. Fourthly, after annealing at 1250°C and quenching in water, ageing occurs during cold rolling and cannot be caused by subsequent exposure at an elevated temperature. Interestingly, static ageing was mainly used to strengthen the Invar alloy in most previous studies [8–12,17–19]. It was only in [20] that the formation of VC and Mo_2C nanoparticles during cold deformation and dynamic ageing was noted.

To achieve a high level of mechanical properties in the Invar alloy, it was initially assumed that a homogeneous, fine-grained structure would form during hot forging at a high temperature. Such a microstructure was obtained. However, this resulted in a significant growth of carbides, which could not be eliminated during subsequent processing. Strong carbide enlargement during hot forging decreases the strength of the Fe-32Ni-2.7(Ti, V, C) Invar alloy since carbides are harder and play a strengthening role. However, large carbides are poorly dispersed in the matrix and are less effective in preventing the dislocation movement. Therefore, to fully realize the dispersion hardening potential of the new Invar alloy, the ingot must be hot worked to breakdown the cast microstructure, and a sample must be obtained for subsequent cold rolling to avoid carbide enlargement. In addition, it is necessary to find a way to dissolve carbides during high-temperature exposure prior to quenching. This ensures that as many dispersed carbides as possible form during subsequent dynamic or static ageing. Another unresolved issue is the scale factor, a critical parameter in transferring the result from laboratory research to the industrial production of the new Invar alloy. In [21], it was shown that the main cause of this issue is the slower cooling rate of a large industrial ingot during casting. This creates an initially coarse microstructure that cannot be corrected as effectively through subsequent hot working and heat treatment as with a small ingot. To minimize this negative impact, processing conditions must be optimized to take into account the initial coarse structure of a large ingot.

5. Conclusions

Studies have shown that the new Fe-32Ni-2.7(Ti, V, C) Invar alloy, which is alloyed with Ti, V and C, demonstrates a high strength (σ_{UTS}=1185 ± 3 MPa) after deformation and thermal treatment, while maintaining acceptable ductility (δ = 4.6 ± 0.3%) and a low CTE (approximately α = 1.2 × 10^{-6} K^{-1}). The significantly higher properties of the new Invar alloy compared to the binary alloy can be explained by the fact that dispersion, solid solution, deformation and grain boundary hardening mechanisms were all realized. On the other hand, the potential of dispersion hardening was not fully realized due to carbide coarsening during hot forging and incomplete dissolution during further annealing.

Future efforts will focus on optimizing hot working and heat treatment in order to suppress carbide growth and enhance the strength of the alloy. These improvements will enable the new Invar alloy to be used in advanced sectors that require high precision and dimensional stability, as well as the ability to withstand heavy mechanical loads.

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