



**KAUNAS UNIVERSITY OF TECHNOLOGY
MECHANICAL ENGINEERING AND DESIGN FACULTY**

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**INVESTIGATION OF TRANSFORMATION PLASTICITY EFFECT
ON QUENCHED AND TEMPERED ALLOY STEEL**

Final project for Master Degree

Supervisor

Assoc. Prof. Dr. Rasa Kandrotaitė Janutiene

KAUNAS, 2015

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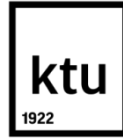
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INVESTIGATION OF TRANSFORMATION PLASTICITY EFFECT ON QUENCHED
AND TEMPERED ALLOY STEEL

DECLARATION OF ACADEMIC HONESTY

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**MASTER STUDIES FINAL PROJECT TASK ASSIGNMENT
Study programme MECHANICAL ENGINEERING**

The final project of Master studies to gain the master qualification degree, is research or applied type project, for completion and defence of which 30 credits are assigned. The final project of the student must demonstrate the deepened and enlarged knowledge acquired in the main studies, also gained skills to formulate and solve an actual problem having limited and (or) contradictory information, independently conduct scientific or applied analysis and properly interpret data. By completing and defending the final project Master studies student must demonstrate the creativity, ability to apply fundamental knowledge, understanding of social and commercial environment, Legal Acts and financial possibilities, show the information search skills, ability to carry out the qualified analysis, use numerical methods, applied software, common information technologies and correct language, ability to formulate proper conclusions.

1. Title of the Project

Investigation of transformation plasticity effect on quenched and tempered alloy steel

Approved by the Dean 2015 y. May m. 11 d. Order No. ST17-F-11-2

2. Aim of the project

The main aim of the work is to determine the influence of bending stress on transformation plasticity effect on quenched and tempered alloyed steels

3. Structure of the project

Introduction, literature review on transformation plasticity of steels, martensitic transformation in steels and factors affecting transformation plasticity, manufacturing of specimens, experimental method includes bending, metallographic and hardness tests, investigation of transformation plasticity of steel during quenching and tempering, effect of austenizing temperature on the quenched hardness, analysis on autodeformation effect during tempering of quenched and bent specimens

4. Requirements and conditions

To prepare final project according to KTU regulations and requirements

5. This task assignment is an integral part of the final project

6. Project submission deadline: 2015 June 1st.

Given to the student Karthikeyan Velliyampalayam Palanisamy

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SUMMARY

This work presents the investigation of transformation plasticity of alloyed steel with 0.39 % carbon content alloyed with chromium (5.3 %) and with considerable amount of molybdenum (1.30 %), silicon (1.0 %) vanadium (0.90 %). Transformation plasticity is a permanent strain observed when metallurgical transformation take place even under small stress applied externally lower than the yield stress of weaker phase. The results of transformation plasticity of the examined steels were effectively compared with three different alloyed steels 40X13, 20X13, 95X18 (GOST 5632-72) of higher chromium content to show evidently that the performance of steel during martensitic transformation directly depends on both internal and external factors such as chemical composition, austenizing and tempering temperature. This work deals with the investigation of evolution of steel structure affected by change in phase composition, stresses, transformation plasticity, quenching and tempering temperature. The steel specimens were heated at a temperature range of 900-1050 °C and then bent during air quenching. Bending stress was 100 MPa. Plastic deflections were observed through all cooling process till room temperature. After quenching the samples were carried out in a thermal processing quality control. Hardness test was carried out for the quenched and tempered alloyed steels specimen to spot the secondary hardening phenomenon. There will be change in density and strength during phase transformations which leads to development of stresses internally in the metals. Different effect of compression and tensile stresses on transformation plasticity of specimens was determined. Tempering of quenched and bent specimens reveals an interesting phenomenon of auto deformation effect which related with loss in deflection and in some cases, increase in deflections of specimens.

Keywords: steel, transformation plasticity, quenching, tempering, martensitic transformation.

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SANTRAUKA

Darbe pristatomas plieno, turinčio 0,39 % anglies ir legiruoto chromu (5,3 %), molibdenu (1,3 %), siliciu (1,0 %) ir vanadžiu (0,90 %), virsminio plastiškumo tyrimas. Virsminio plastiškumo efektas – tai ilgalaikė deformacija, susidaranti metalurginių virsmų metu, kai išoriniai įtempiai neviršija silpniausios fazės takumo ribos. Lyginant tiriamo plieno virsminio plastiškumo rezultatus su kitomis skirtingai legiruotomis plieno markėmis 40X13, 20X13 ir 95X18 (GOST 5632-72), turinčiomis įvairių legiravimo elementų kieki, aiškiai matyti, kad plieno gaminių elgsena martensitinio virsmo metu labai priklauso nuo tokių faktorių kaip cheminė sudėtis, grūdinimo bei atleidimo temperatūros. Šiame darbe pristatomas plieno struktūros evoliucijos tyrimas, kai pliene veikia faziniai virsmai, išoriniai įtempiai, plienas kaitinamas grūdinimui ar atleidimui, pasireiškia virsminis plastiškumas. Plieno bandiniai buvo kaitinami iki temperatūrų 900-1050 °C, tada lenkiami aušinant ore. Lenkimo įtempiai bandiniuose sudarė 100 MPa. Viso aušinimo metu buvo matuojamas lenkiamų bandinių plastinis įlinkis. Po grūdinimo ir po atleidimo įvairiose temperatūrose buvo matuojamas bandinių kietumas – užfiksuotas antrinis kietėjimas. Buvo nustatytas skirtingas tempimo ir gniuždymo įtempių poveikis virsminiam plastiškumui. Atleidimo metu buvo pastebėtas autodeformacijų efektas, kai lenkti bandiniai tiesinasi arba jų įlinkis, gautas po grūdinimo, didėja dar labiau.

Raktiniai žodžiai: plienas, virsminis plastiškumas, grūdinimas, atleidimas, martensitinis virsmas

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1. INTRODUCTION

Transformation plasticity is the irreversible deformation behaviour of transforming specimens even under a load stress-state with an equivalent stress significantly lower than the yield stress [1]. Transformation induced plasticity has wide range of practical situations such as steel quenching or welding, increasing the toughness of materials using transformation induced plasticity steels, etc. It has a favourable influence on the overall mechanical characteristics of carbon steels due to the two involved mechanisms, i.e., plastic flow and martensitic transformation [2].

Many theoretical investigations and experiments [1-7] have been prepared for improved understanding on the phenomenon of transformation plasticity but still it is not fully explained yet due to several different factors which tend to affect the plasticity transformation. Some of the major internal factors which have great influence on the transformation plasticity are; the chemical composition of the steel which plays an import role in evolution of martensitic transformation especially the amount of carbon content available and other important alloying elements such as molybdenum, vanadium, chromium etc.

External factors like austenizing temperature, quenching temperature and tempering temperature also play an efficient role in determining the behaviour of plasticity transformation and hardness of the steel.

The steel used for the investigation is THG2000 steel alloyed with chromium, molybdenum and vanadium. The mentioned steel has wide range of applications in indexable drills, milling cutters, and transmission parts for automobiles due to has high resistance to abrasion at both high and low temperature and resistance to thermal fatigue as well. For the investigation of influence of alloying elements different alloyed steels 40X13, 20X13 and 95X18 (GOST 5632-72) are chosen for experiments for having effective comparison.

The scientific works were mostly found explaining the micromechanical modelling of transformation plasticity [1, 2, 7, 13, 23]. Only very few scientific works deals with the carbide forming and influence of stress on microstructure of steel during tempering [15, 19]. But there are lack of scientific works which explains the direct importance of influencing of alloying elements on transformation plasticity.

The **main aim** of the work is to determine the influence of bending stress on transformation plasticity effect on quenched and tempered alloyed steels. Transformation plasticity caused due to martensitic transformation during quenching and tempering of steels as steel possess highest plasticity during martensitic transformation. Different steel grades are used in experiments for comparison. The experimental method includes bending test, metallographic test and hardness test. The important **tasks of the work** is to determine,

- the influence of chemical composition (content of carbon and other alloying elements like chromium, molybdenum and vanadium) on phase contents of martensite and retained austenite under the concern of heat treatment.
- the effect of austenizing temperature on hardness, plastic deflections and martensitic start temperature of quenched and tempered alloyed steels.
- the autodeformation effect which is one of the most interesting phenomenon related with change in deflection of the specimen during tempering of bent and quenched specimens.

2. LITERATURE REVIEW

2.1. GREENWOOD-JOHNSON AND MAGEE EFFECT

The mechanism for plastic transformation was explained by the theories of Magee [3] and Greenwood–Johnson [4]. Two physical interpretations of this phenomenon were given by Greenwood and Johnson (1965) for the case of diffusion transformation and by Magee (1966) for the case of martensitic transformation [5].

Greenwood and Johnson [4] had assumption that the weaker phase could deform plastically for a plastic material to accommodate the internal stress due to volumetric change associated with the phase transformation and applied external stress [6]. A strain induced transformation plasticity analytical equation based on a flow theory and the Levy–von Mises equation was derived [6].

Two mechanisms are considered to explain transformation plasticity from microstructural point of view and are as follows,

1. The Magee (1966) mechanism corresponds to the formation of selected martensitic variants resulting from the applied stress [7].
2. The Greenwood–Johnson (1965) mechanism corresponds to the micromechanical plastic strain arising in the parent phase from the expansion of the product phase [7].

The importance of these two mechanisms depends on the material and the transformation under consideration. Both mechanisms are generally present in diffusional and diffusionless transformations [7].

Greenwood and Johnson proposed a pseudo-creep theory for transformation plasticity according to which plastic flow during the phase change results from an interaction between the relatively low external stress and high randomly oriented internal stresses caused by the difference of the specific volumes of the two phases [7].

The Greenwood-Johnson effect describes the difference in volume among two co-existing phases which also induces the yielding in the weaker of the two phases [8]. Hence the plastic flow is permitted in spite of having insufficient externally applied load to induce the plasticity in the material. Phase transitions were also addressed by Magee (1969) [10], who showed that external loading can trigger martensite formation in preferred directions which

cause orientation effect influencing the macroscopic shape of the loaded body [8]. The Greenwood-Johnson mechanism and the Magee effect [3,4] indicates that the material undergoing martensitic phase transition has both volumetric and as well as deviatory response with directional dependency [8].

Presently, the Greenwood and Johnson's constitutive comparison was adjusted and stretched out by numerous different research analysts [11-15]. This phenomenon is effectively carried on by the plastic change accompanied with the transformation induced volume and shape change of micro regions in the parts of a grain. The corresponding micro stress states must be in global equilibrium with the load stress state [1].

2.2. ANALYSIS OF TRANSFORMATION PLASTICITY

The major factors that affects the transformation plastic deformation in the present calculations can be classified into (a) an externally applied stress, (b) yield stress of each phase, and (c) ferrite to austenite yield stress ratio [16]. The existence of transformation plasticity increases the evolution of non-isotropic strain, which states that the plastic strain besides the stressed axis [17]. While the non-isotropic strain can be calculated from the difference between the observed length change and that expected from the isotropic transformation strain as shown schematically at Fig. 2.1 [17].

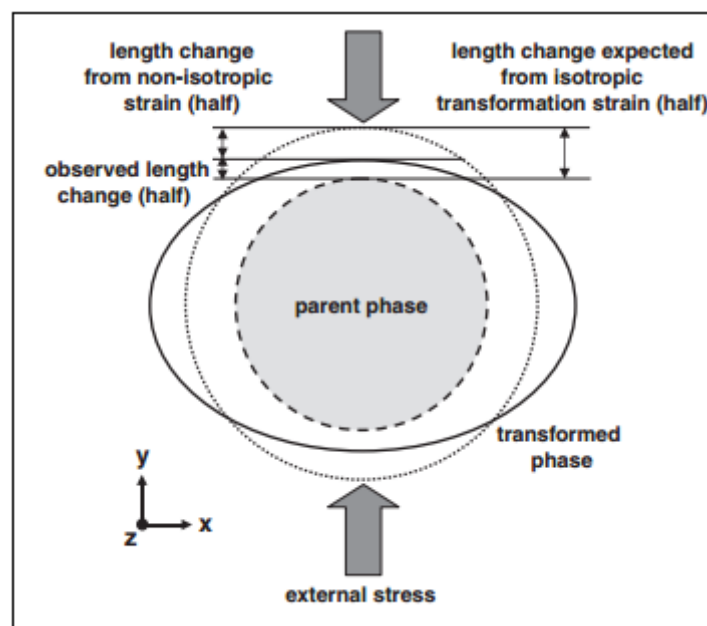


Fig. 2.1. Length change from non-isotropic strain during transformation accompanied by volume change [17]

The development of non-isotropic strain is widely engaged by transformation plasticity, when the transformation kinetics is faster, it ignores the contribution of creep in austenite and ferrite but when transformation kinetics is slow, it is affected by creep in austenite and ferrite phases.

2.3. DEFORMATION INDUCED MARTENSITIC TRANSFORMATION IN STEELS

The martensitic transformation is induced by deformation in metastable austenitic steels. This is called the deformation induced martensitic transformation. The role of applied stress and plastic strain on the martensitic transformation plays a vital role in order to understand the nature of phenomenon [18]. *I. Tamura* proposes that the deformation-induced martensitic transformation can be understood only in terms of the effect of applied stress rather than the effect of strain [19]. An outstanding increase in elongation was obtained when martensite is formed at the time of deformation.

When the austenite is deformed at temperatures above martensitic start temperature M_s^σ (e.g. at T_I in Fig. 2.2), the austenite starts to deform plastically at a stress σ_a , and is strain hardened up to σ_b and then the martensitic transformation begins. This decrease ($\sigma_c - \sigma_b$) of critical applied stress for martensite formation is due to the plastic deformation of austenite. The strain-induced martensite nucleation proposed by *Tamura et al., 1972* [20], and the stress which is locally concentrated at obstacles (e.g. grain boundaries, twin boundaries, etc.) by the plastic deformation of austenite, and thus the concentrated stress becomes equivalent to σ_c in Fig. 2.2 These are the two different views in the role of plastic deformation of austenite [18].

The metastable austenitic phase transforms into martensite at room temperature which appears under different crystallographic structures. In the case of transformation plasticity in steels, the α -martensite with body-centered cubic structure, appears during plastic deformation which is highly responsible for the high strength providing outstanding ductility [21]. As in case of Fe- based shape memory alloys, the formation of α -martensite with hexagonal-packed cubic structure which is mainly accountable for the perfect shape recovery [21].

Austenite-to-martensite transformation occurs at temperatures above the martensite start temperature (M_s) when the summation of the mechanical energy due to the externally applied stress and the chemical driving force exceeds a critical value [6]. This transformation

can be induced via stress-assisted nucleation at the similar nucleation sites which are highly responsible for the thermal martensitic transformation during cooling [22].

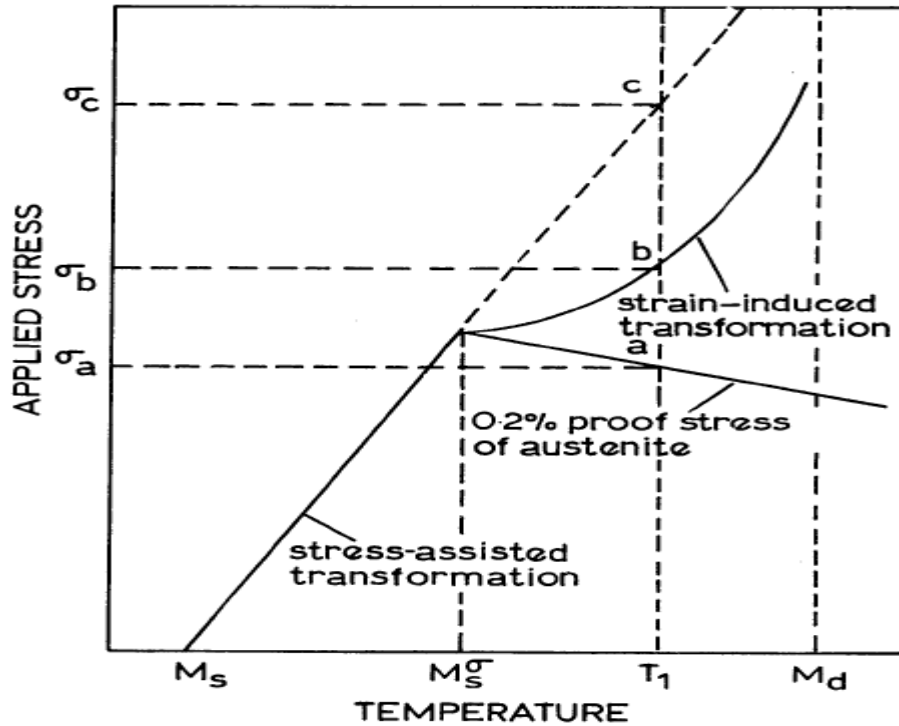


Fig. 2.2. Critical stress for martensitic transformation as a function of temperature [18]

Martensitic transformation has wide range of attention due to its large potential applications as it provides superior mechanical properties for the product which is transformed, especially the strength of martensite and the corresponding thermomechanical treatments to produce adequate behaviour for engineering applications [23].

Shape memory alloys (SMA) and steels exhibits enhanced responses to transformation-induced plasticity and these are the two examples of the significant roles martensite formation can play [8].

2.4. SEVERAL FACTORS AFFECTING TRANSFORMATION PLASTICITY

2.4.1. Grain size

With decreasing austenite grain size, the maximum elongation increases and the elongation peak shifts to lower temperatures and becomes broader [18]. With increasing austenite grain size, the maximum elongation decreases and the elongation peak shifts to higher temperatures and becomes narrower as shown in Fig. 2.3 [18].

The transformation plasticity in steels are determined by the orientation of the grain with respect to the loading. The size of the retained austenite grains controls the transformation rate. When the austenitic grain size decreases the martensitic transformation increases [8]. The grain size of the retained austenite can be controlled through a suitably chosen thermal processing route to some extent [24].

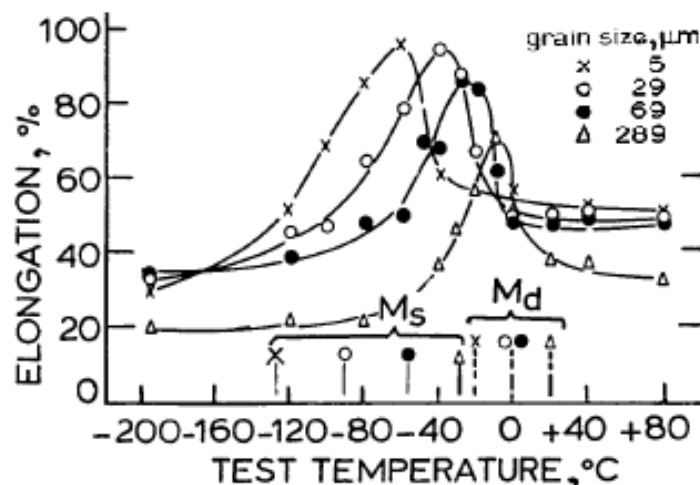


Fig. 2.3. Elongation based on grain size and temperature [18]

The mechanical response of transforming retained austenite grains powerfully depends on the grain size in case of multiphase carbon steel. The surface energy term in the transformation model captures the increased resistance to transformation due to a reduction in the size of the austenitic grains [25].

2.4.2. Strain rate

- ❖ The effect of strain rate plays a vital role as a factor affecting transformation plasticity. When specimens are deformed in air or in a gas atmosphere, transformation plasticity occurs less with increase in strain rate readily because of its lower transformation rate due to the adiabatic heating of the specimen [19].
- ❖ In other case, when specimens are deformed in water or in a liquid atmosphere, the decrease in tensile elongation is found to be very little with expansion in higher strain rates it may be due to the elimination of temperature rise of the specimen by quick removal of specimen heating with the liquid atmosphere [19].

2.4.3. Alloying elements and their related factors

The transformation plasticity phenomenon is directly proportional to the effect of alloying elements and the carbon content present in a steel. The alloying elements affects the stability of austenite, martensite morphology, and the temperature difference between M_s and M_f and the diffusion rate of carbon during plastic transformation [19].

Alloying elements vary not only the habit plane and the transformation behaviour of martensite but also the martensite morphology. Steels exhibit lenticular martensite, e.g. Fe-high-C and Fe-high-Ni-C steels [19].

Carbon Influence on Transformation Plasticity

With an increase in the carbon content, the martensite hardness is increased remarkably, although the austenite hardness is only slightly increased, as shown in Fig. 2.4. Work hardening can be obtained by a small amount of martensite formation in high-carbon steels [19]. Transformation plasticity possess larger uniform elongation.

Alloying elements may be divided into two groups based on their distribution in steel,

- Elements that does not form carbides in steel (e.g. Al, Cu, Ni, Si, Co and N)
- Elements that form carbides in steel (e.g. W, V, Ti, Cr, Mn, Mo, Zr and Nb) [43].

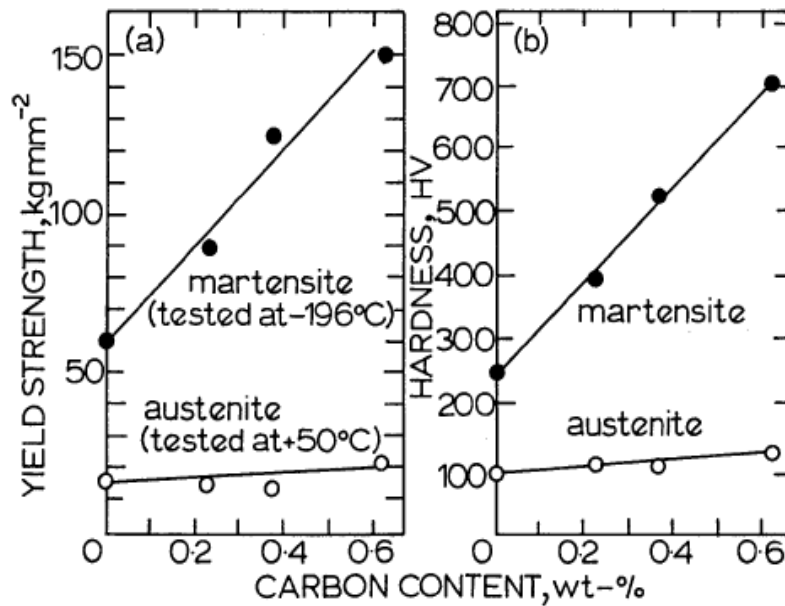


Fig. 2.4. Changes in yield strength (a) and hardness of martensite (b) with C content in Fe-Ni-C alloys [19]

The austenite to martensite transformation directly depends on the amount of carbon level in the steel and it also depends on the yield strength of the material. The steels with higher carbon content possess stable retained austenite this can be due to the phenomenon that the transformation of these austenites are not obtained immediately.

The strain level at which the retained austenite starts to transform to martensite is determined by the available amount of carbon content in steel. Upon deformation retained austenite begins to transform almost immediately for lower carbon levels increasing the work hardening rate. At higher carbon contents, the retained austenite is more stable and begins to transform only at strain levels outside those produced during forming [26, 27].

The martensitic start temperature is directly proportional to the amount of carbon content available. For lower amount of available carbon content, the martensitic transformation tends to start at very high temperatures but in case of higher carbon content the martensitic start temperature is comparatively lower as shown in Fig. 2.5.

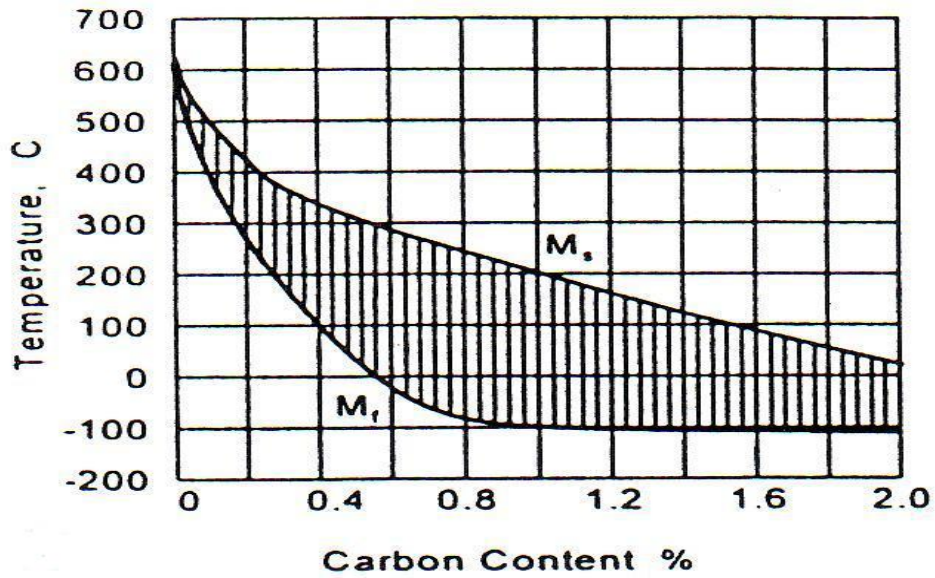


Fig. 2.5. Effect of carbon content on martensite transformation [28]

Most alloying elements that enter into solid solution in austenite lower the martensite start temperature (M_s), with the exception of Co and Al as shown in Fig. 2.6 [43]. It is also evident that the strong carbide forming elements like vanadium, molybdenum and chromium also have an impact on martensitic transformation start temperature [42].

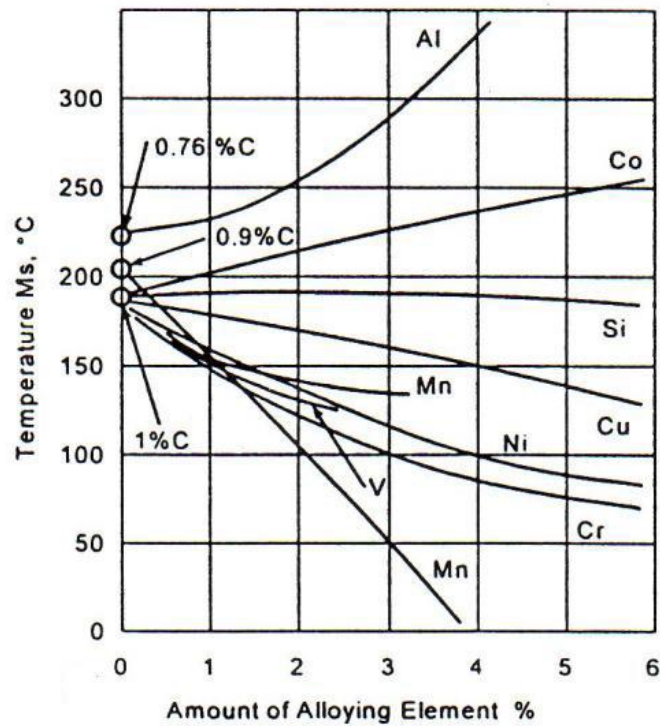


Fig. 2.6. Alloying elements at 1 % C on the martensite transformation [42]

Silicon

In recent years, addition of Si content to steels are lowered in the aim of reducing the surface quality, industrial casting and galvanising problems. Addition of silicon increases strength, decreases the weldability. Silicon substitutes as a ferrite stabiliser helps to retain carbon-enriched austenite by suppressing cementite precipitation from austenite [29, 50]. Silicon is also a strong austenite former agent providing finer grain size due to the nucleation of austenite grain in different sizes.

Manganese

Addition of manganese increases the strength, shock resistance, toughness, hardenability, weldability, hot formability, and eliminates change in ductility. In addition, Mn is a strong austenite former by reducing the eutectoid temperature below to room temperature [28]. An increase in manganese may compensate for any reduction in silicon [30], limiting the amount of bainite that can form as it lowers carbon concentrations. Pronounced banding may occur in steels containing a large manganese concentration [31].

Aluminium influence on transformation plasticity

The less addition of Al affects the chemical structure of the alloys. There are advantages to this phenomenon for example, a fully martensitic structure cannot be produced in the heat-affected zone of a resistance spot weld. The steel also depends more on aluminium rather than silicon to suppress the formation of cementite [29], which helps avoid the problem of the adherent fayalite scale that forms on the surface during hot-rolling [32,33]. Aluminum (Al) is used as a grain refinement agent particularly in the form of AlN particles. Aluminium obstructs cementite precipitation and hence can substitute for silicon [19]. Steels in which silicon is replaced by aluminium may therefore be weaker due to the fact that Al does not strengthen ferrite [34].

Phosphorus

Addition of phosphorous decreases the toughness, impact resistance, cold formability, weldability and increases corrosion resistance. Its content is limited 0.035% max in quality steels. Phosphorous, on the other hand, strengthens ferrite [35].

Molybdenum

Molybdenum is a solid solution strengthener of ferrite and retards pearlite formation. Mechanical properties of low-silicon steels can be enhanced comparably to high-silicon steels

by addition of molybdenum, ultimate tensile strength in excess of 1000 MPa with a total elongation of about 36% [36].

Zirconium (Zr), Titanium (Ti), and Tantalum (Ta)

These alloying elements are strong carbide formers even better than Cr. Therefore they are commonly used in austenitic stainless steel to free the Cr and enhancing the corrosion resistance. These elements easily forms small carbides at grain boundaries which delivers very fine grain size as a result providing high strength and ductility for low alloy steels which are commonly used in automotive industry.

Effect of Nb on the stability of retained austenite

Niobium in solid solution increases the amount of retained austenite which in turn also increases the hardenability of the steel [37]. Retained austenite in Nb steel exhibited a higher stability, resulting in the slow transformation kinetics of retained austenite to martensite and hence the effect of transformation plasticity is comparatively weak during tensile straining [38].

Steel with Nb has higher Yield strength and tensile strength when compared with Nb-free steel, which is mainly due to the smaller size of ferrite grains, the higher volume fraction of bainite and their ability to precipitate Nb in the multi-phase microstructure of Nb steel. The uniform elongation and the total elongation of Nb steel were lower than that of Nb-free steel [38].

Copper

Copper is an austenite stabiliser which helps to retain austenite [39]. According to *Kim, S. J et al., 1991* [31]. Copper can boost the overall strength and hence it can be an option for replacing silicon in the ways of retaining austenite and increasing strength of ferrite which is restricted to max. 0.35%.

Chromium

Increase in chromium content increases, strength, hardenability, resist to corrosion, and decreases the oxide formation tendency. Chromium obstructs the dislocation motion particularly at elevated temperatures as it is a strong carbide former. Higher critical temperatures is obtained in in heat treatment for chromium steels.

During martensitic transformation steel possess kinetic plasticity. When steel is hardened at the optimal hardening temperature relative rate of the kinetic plasticity of chromium steels (Cr content from 0.99 % until 18.20 %) reaches maximum [40].

The Fig.2.7 shows that kinetic plasticity of low alloyed steel with 0.99 % of Cr increases with increase in hardening temperature and do not decreases after overheating (temperature 3). Overheating temperature is very important for properties of high chromium stainless steels (12.89 % and 18.20 % of Cr) as its relative rate of kinetic plasticity is directly proportional to overheating [40]. Quantity of retained austenite are formed when overheated which is one of the major reason for the decreases in the relative rate of kinetic plasticity.

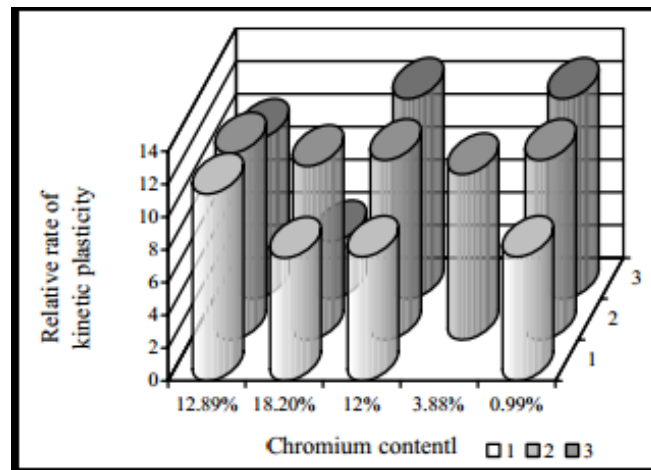


Fig. 2.7. Dependence of chromium content on kinetic plasticity. 1. Hardening temperature lower than optimal, 2. Optimal hardening temperature, 3. Hardening temperature higher than optimal [40]

All alloying elements that form solid solutions in ferrite affect its hardness and chromium possess major effect on ferrite hardness as well [28]. The hardness increase was caused by substitutional solution as shown in Fig. 2.8 where Cr gives the smallest hardness increase when compared with other alloying elements. The ferrite hardness increase directly be influenced by the concentration of alloying elements used. Cr is a most convenient alloying element in steel that is to be processed by cold working in which good hardenability is required [28].

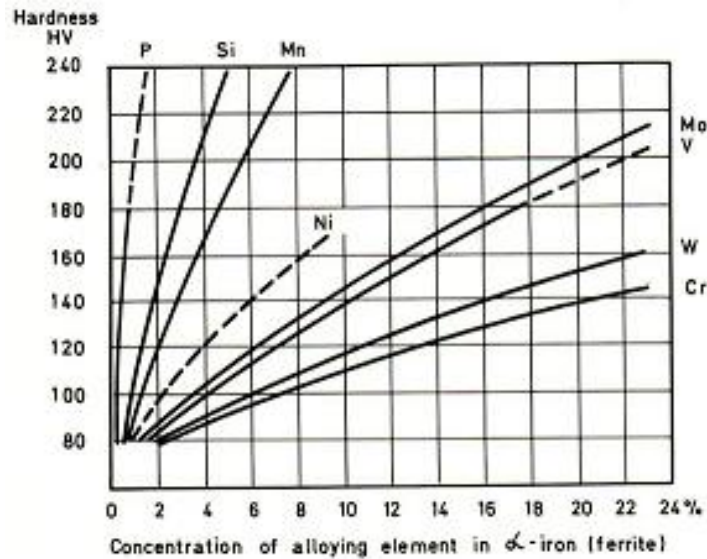


Fig. 2.8. Effect of substitutional alloying element additions on ferrite hardness [28]

2.5. TRANSFORMATION PLASTICITY ON OTHER MATERIALS

The studies on plasticity transformations have been conducted on many systems including steels, rare earth metals, commercially pure titanium, zirconium, cobalt and both transformations of uranium and so on. During welding and quenching operations in steel, some solid state structural transformations may take place [28].

2.5.1. Transformation-Induced Plasticity of Expandable tubular materials

Transformation induced plasticity plays an important role in tubular expandable materials as these materials are widely used in oil industries which possess major characteristics such as; high plasticity, high strength and high work hardening rate [41]. The strength increases along with the plasticity for expandable tubular materials, when the austenite transforms to martensite the transformation plasticity mechanism is induced during the time the material is subjected to external force [41]. These alloys possess high strengthening-plasticity with expandable performance which are exceptional [41]. Expandable alloys with excellent expandable performance lies in the hatched zone showed at Fig. 2.9.

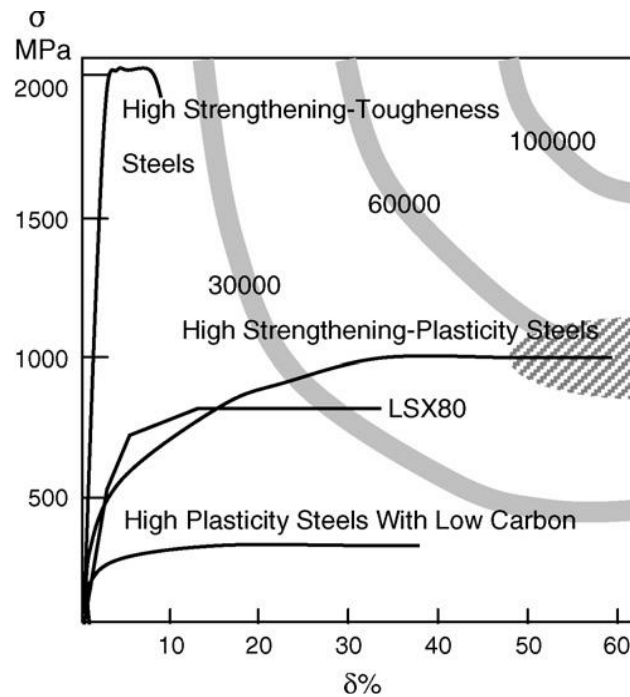


Fig. 2.9. The comparison of strengthening-plasticity among various types of steels [41]

2.5.2. Transformation Plasticity of Titanium

An increase in the creep rate is observed during the transformation on titanium [9]. For Ti-6Al-4V the transformation strain and applied stress are proportional upto 400 psi in tension, the transformation strain vs stress curve slope should be in agreement with the Greenwood and Johnson [4] theoretical predictions [9]. In pure titanium which is used commercially, the linear relationship between plastic flow and applied stress plays an important role in determining the transformation plasticity [9].

Decrease in the mechanical properties are obtained such as, decrease in area, elongation, fracture strength of titanium as the transformation plasticity effect is increased, this may be due to the phenomenon that transformation plasticity increases by increasing the cycling stress or by the number of temperature cycles which increases the grain size during transformation plasticity [9].

2.5.3. Transformation-induced plasticity in NiTi shape memory alloy

Transformation-induced plasticity was effectively observed in NiTi shape memory alloy. The austenite phase of a solutionized Ni-Ti alloy shows a serrated stress-strain curve in range of 311–328 K, below the temperature at which the deformation mechanism of the

austenite phase changes from that of stress-induced martensitic (SIM) transformation in the low temperature ranges to their slip at high temperature range (329 K) [38].

2.5.4. Transformation plasticity of rare earth ortho-phosphates

Transformation plasticity of rare-earth orthophosphates (TbPO_4 and $(\text{Gd}, \text{Dy})\text{PO}_4$) having xenotime phase can be established by using nano-indentation and TEM characterization [43]. The deformation twinning in monazite is very much similar to the martensitic xenotime to monazite phase transformations and which it needs only small shuffles. Evidences are available that these transformations may reverse with time [43]. Earth materials can be weakened by transformation plasticity phenomenon. Large rare-earths such as La and Ce form orthophosphates with the monazite structure. Small rare earth metals such as Y and Lu form orthophosphates with the xenotime structure [43].

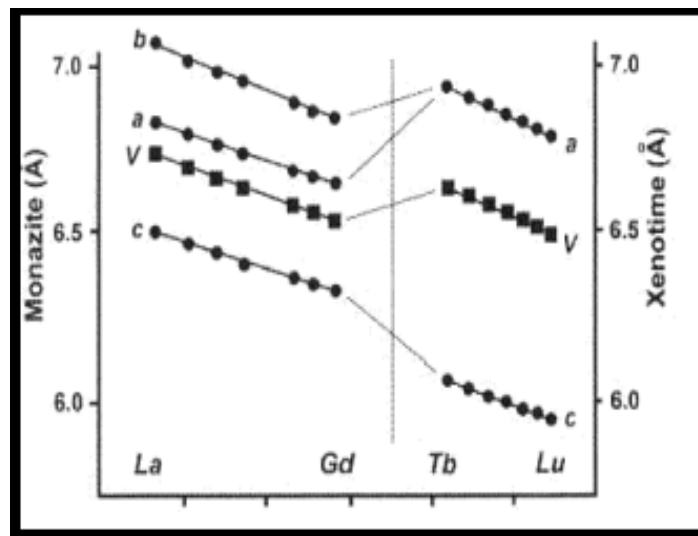


Fig. 2.10. Lattice parameter of the rare earth phosphates [40]

The phenomenon of transformation plasticity possess its application in ceramics which is a brittle field mechanism called transformation weakening which was generally proposed for interphase crack deflection but it is not proposed in the sense of reducing pull-out friction [43].

2.5.5. Transformation zone in Ce-TZP ceramics

In 9Ce-TZP ceramics very large transformation zone was observed which was related with eruption kind of transformation behaviour leads to a discontinuous stepwise transformation plasticity [44].



Fig. 2.11. Transformation Zone [44]

Whereas in ZrO_2 -containing ceramics when it is subjected to loading conditions Stress-induced transformation may induce plasticity localization [44]. During localization the one of the important characteristic feature of stress-strain curves is the strain softening. For any loading conditions it is possible to determine whether the localization may appear or not with indicating the orientation of the localization band also [44, 45].

2.6. APPLICATIONS

Trip steels are one of the most efficient applications of transformation plasticity and it is widely used in recent times. *Zackay et al* have developed ultrahigh strength steel called TRIP steels in which the ductility is enhanced by the transformation plasticity of retained austenite and the strength is increased by the ausformed martensite [46]. Excellent strength-ductility combination of transformation plasticity steels are obtained by the presence of retained austenite. The retained austenite transforms to martensite during plastic deformation. Trip steels have high formability, while retaining excellent strength and makes the trip steels very attractive for structural parts in the automotive industry [27].

Transformation induced plasticity has its major application on armour steel which has a Poisson's ratio of 0.3 and Young's modulus as 190 GPa [47]. Armour steels undergoes evolution of martensitic volume fraction with the increasing of the strain during the uniaxial tension test at various temperatures [47]. The elastic behaviour of austenite starts initially with increasing strain followed by the plastic flow without transformation was found to be occurred, later the plastic flow inside the austenite and martensitic phase transformation was started [47].

3. METHODOLOGY

3.1. MANUFACTURING OF SPECIMENS

The investigation of transformation plasticity of THG2000 Swedish steel during martensitic transformation was carried out which has high resistance to abrasion at both high and low temperature and resistance to thermal fatigue as well. The steel THG2000 is a chromium-molybdenum-vanadium alloy steel. This steel possessed wide range of applications in indexable drills, milling cutters, and transmission parts for automobiles.

Table. 3.1. The chemical composition of steel according to the specification of producer. Wt% [45]

Steel grade	C	Si	Mn	Cr	Ni	Mo	V	Fe
THG 2000	0.39	1.0	0.40	5.30	0.15	1.30	0.90	Bal.

For this investigation on transformation plasticity the specimens were manufactured from the above investigated steel. Specimens with rectangular cross sectional measurement of 6X8X100 mm³ were prepared from a steel rod of diameter, ϕ 11.9mm.

The transformation plasticity phenomenon was investigated during quenching and tempering of alloyed steel specimens. For this investigation different steel grades were selected for the comparison shown in Table. 3.2.

Table. 3.2. The chemical composition of steels used for comparison. Wt%

Steel grade GOST 5632 – 72	Chemical composition %							
	C	Cr	Si	Mn	Mo	Ni	V	Fe
40X13	0.35	13	0.35	0.4	-	0.30	-	Bal.
95X18	0.97	18	0.6	0.6	0.10	0.39	-	Bal.
20X13	0.19	12.71	0.23	0.29	-	0.15	-	Bal.

The soft annealed microstructure of THG2000 alloyed steel consists of a ferrite matrix (white areas) in which carbides (grey particles) are embedded shown in Fig. 3.1. In THG2000 alloyed steel the carbides are chromium (Cr), molybdenum (Mo) or vanadium (V) carbides, depending on the composition of the steel. Vanadium increases the strength of the steel by forming stable carbides. Carbides are mix of carbon and alloying elements and are characterized by very high hardness.

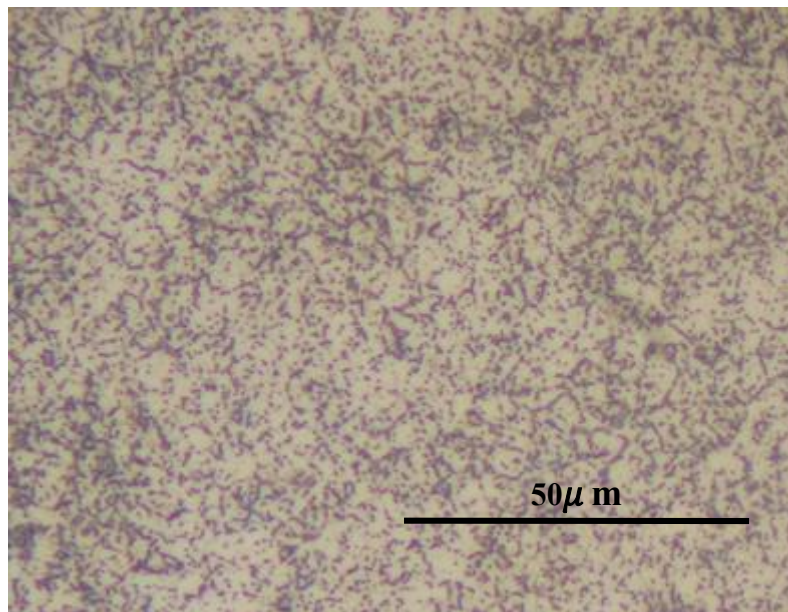


Fig. 3.1. Annealed microstructure of THG2000 specimen

3.2. BENDING TEST

The specimens were austenized in a protective environment ($N_2+CO+CO_2$) at 900°C, 940°C, 980°C, and 1020°C for air quenching. These austenized specimens were further introduced in a special bending device shown in Fig. 3.2 for the transformation plasticity examination and also air cooled. The specimens are subjected to be loaded in bending load generating bending stress of 100MPa which does not exceed the 10 % of yield strength of the material ($\leq 10\% R_{p0.2}$). (Yield strength of THG2000 is 1000MPa for M_s 310°C) [50].

The austenized specimen is carefully placed in-between the specimen supports. The bending load of 100 MPa is applied over the specimen and corresponding deflections are noted

by using the dial gauge attached to the bending device. The readings are taken at different time intervals from 0.45 seconds to 15 minutes.

The plastic deflection is obtained in 0.01mm accuracy throughout the process of cooling till room temperature. Due to the change in temperature elastic deflection is also attained by this phenomenon leads to transformation plasticity.

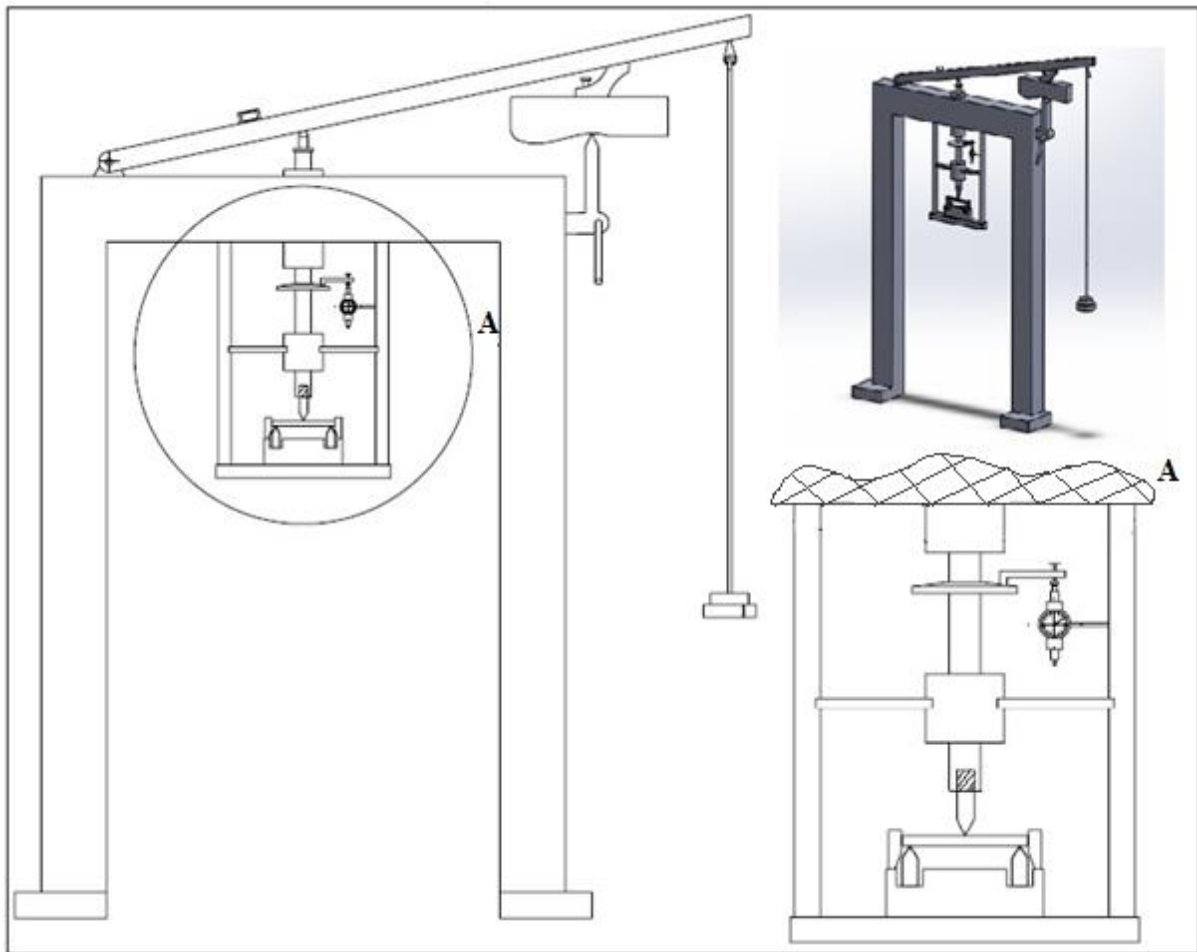


Fig. 3.2. Bending device for the transformation plasticity examination

Highest plasticity possess when the steel is subjected to martensitic transformation. Alloying elements plays a vital role in quenched steel plasticity, especially the carbon and chromium content in the material. The temperature decreasing of a specimen was determined by using a chromel-alumel thermocouple, welded to the specimen. This specimen temperature measurement is highly important to determine the martensitic start temperature of the specimen.

3.3. METALLOGRAPHIC TEST

After quenching the samples were carried out in a thermal processing quality control. The samples were cut by using METKON MICROCUT125 Low speed precision cutter, and the preparation of mould is done by using TECHNOVIT 407.

The investigation of microstructure is carried out by monitoring with laser analyzer LMA Carl Zeiss using a video camera YCH15. The samples for optical analysis were ground, polished with Lam Plan polisher and etched in 3% Nital solution.

3.4. HARDNESS TEST

Rockwell hardness test was carried out for both quenched and tempered specimens. It can be defined as the resistance to indentation. It is determined by measuring the permanent depth of the indentation. It measures the permanent depth of indentation produced by a force/load on an indenter. Rockwell hardness test measurements were obtained for the specimen with VERZUS 750CCD universal hardness meter. For obtaining these measurements average values of three different tests on a sample was carried out.

4. RESULTS AND DISCUSSIONS

4.1. INVESTIGATION OF TRANSFORMATION PLASTICITY OF SPECIMENS DURING QUENCHING

Highest plasticity is possessed by steel when it undergoes martensitic transformation and the plasticity obtained is greater comparing to the one obtained after tempering. The plasticity value of the quenched steel is mainly affected due to its carbon content and related factors such as type of quenching, austenizing temperature, tempering temperature, content of alloying elements, etc.

The carbon content and alloying elements which impact the relation of phase contents of martensite and austenite under effect of heat treatment, and the influence of stress on phase and structural transformations with respect to their quantitative ratio and the volume differences during quenching and tempering are the most essential factors in determining transformation plasticity.

The effect of stress also plays an important role as the austenite percentage is higher at lower stress levels [50]. Due to effect of stress, the dislocations are formed and redistributed which enhances the formation of carbides and the migration of chromium and carbon towards dislocations. The deformation caused due to stress along with tempering improves macro and micro plastic deformations, resistance to relaxation etc., which in turn increasing the performance of steel [51]. Hence the morphology of the isolated carbide depends on the acting stress direction.

It is highly required to have knowledge on kinetics of transformation plasticity patterns of different steel grades and its heat treatment methods in steel production in order to make efficient use of transformation plasticity effect. As transformation plasticity is known to play an important role in steel making processes. For instance, in heat treatment process the shape of the product and residual stress are affected by transformation plasticity.

When the specimen is in contact with loading and atmospheric air cooling, the specimen is able to resist the bending force applied which does not exceeds the 10 % of yield strength of the material.

It is important to note the phenomenon that austenizing temperature places a critical role in start time period of martensitic transformation of quenched specimens. From Fig. 4.1 it is clear that austenizing temperature and time the martensitic transformation begins are directly proportional, the other phenomenon is that plasticity transformation of the specimen is also directly proportional to the austenizing temperature of the quenched specimen as in Tq. 1020°C the plasticity transformation starts at 4 minutes and for Tq. 900°C it starts prior around 3 minutes.

The martensitic transformation starts about 370°C for lower austenizing temperature (940°C) and decreases to around 310°C as shown in Fig. 4.1 for increasing austenizing temperature (980-1020 °C) as austenite enriches with carbon and chromium which happens mainly because chromium carbides solves in matrix. Which is highly evident that austenizing temperature and carbon content plays a critical role in determining martensitic transformation start temperature.

THG 2000 steel is theoretically compared with 4X5Mφ1C GOST 5950-73 steel of similar chemical composition shown in Table. 4.1, which possess alike martensitic start temperature around 305°C [52], which gives us the indication that the chemical composition especially the amount of carbon content also plays significant role in determining the start of martensitic transformation as both the steels with similar chemical composition possess similar martensitic start temperature.

Table. 4.1. The chemical composition of steel. Wt% [52]

Steel grade	C	Si	Mn	Cr	Mo	V	Fe
4X5Mφ1C GOST 5950-73	0.37-0.44	0.90-1.20	0.20-0.50	4.50-5.50	1.20-1.50	0.80-1.10	Bal.

Martensitic transformation is a non-diffusional transformation and hence all the carbon dissolved in face centred cubic austenite lattice remains in the new phase - martensite lattice saturated with carbon. It is commonly said that Magee effect plays a vital in martensitic transformation [8].

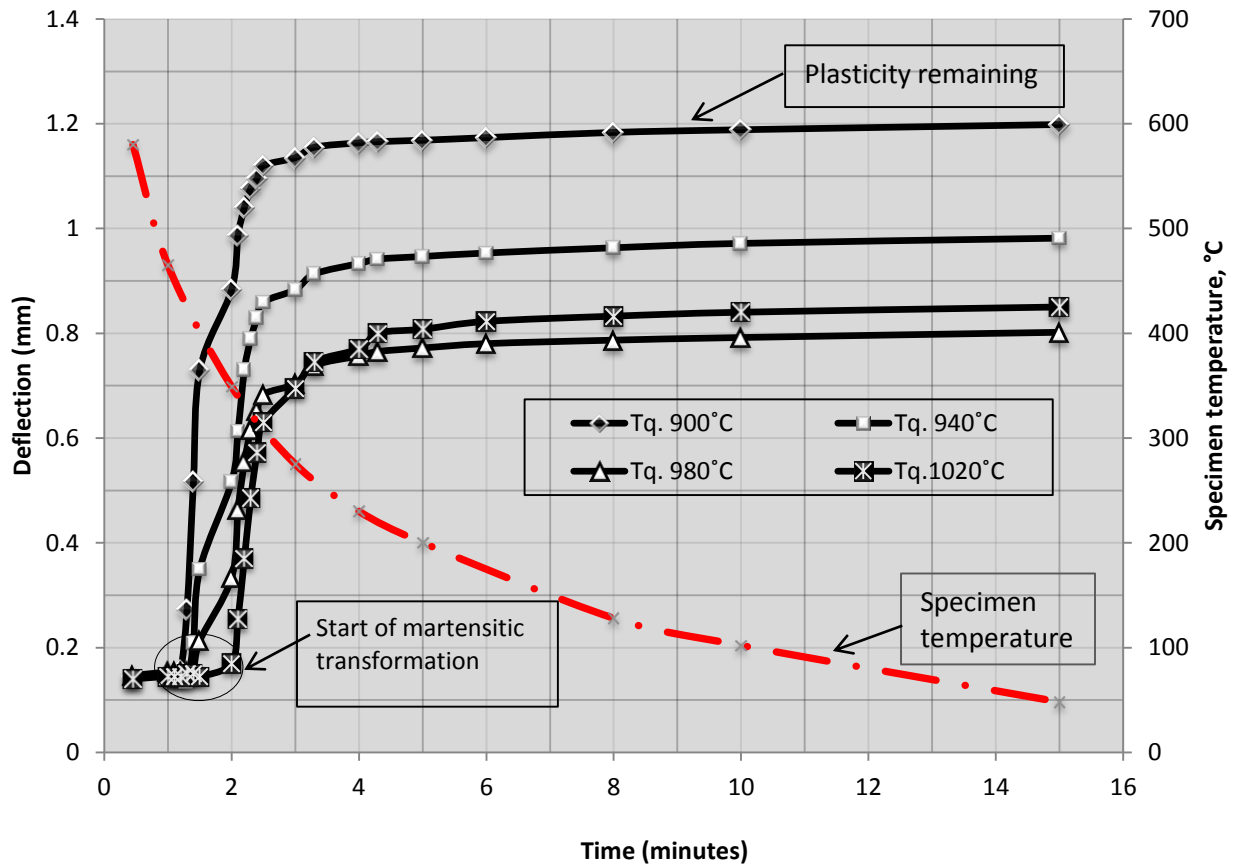


Fig. 4.1. Plastic deformation of THG2000 steel specimens air quenched from different austenizing temperature

The influence of alloying elements on transformation plasticity plays a vital role as they have higher influence in the progress of martensitic transformation as shown in Fig. 15. The plastic deformation of different steel specimens which are air quenched from higher austenizing temperature 1020°C and 1050°C are evident to prove that the composition of alloying elements affects transformation plasticity.

During quenching of specimens after heating at austenizing temperature, the martensite transformation does not begin immediately. It is to be noted that the martensitic transformation begins around 2 minutes at higher austenizing temperature for all different steel specimens used for comparison, which is evident that increasing the austenizing temperature also plays an important role in start of martensite transformation. The higher the temperatures applied to specimens, the greater the deformations obtained.

Less plasticity is obtained in THG2000 steel when compared with 40X13, 95X18, 20X13 steels as shown in Fig. 4.2. This may be due to the presence of vanadium content in THG2000 steel. As vanadium is a strong carbide former it also determines the size of grains especially smaller grain size which enhances stable deflection, which is evident that the plasticity transformation of the specimen is directly proportional to the chemical composition of the quenched specimen. The presence of carbon and chromium content has a significant role as it determines the plasticity transformation of the specimen, which is apparently shown in Fig. 4.2. The amount of carbon determines the strain level at which the retained austenite begins to transform to martensite. The retained austenite begins to transform almost immediately upon deformation at lower carbon levels and it is more stable at higher carbon contents [27].

The temperature of martensitic transformation (beginning and end) depends on the presence of carbon content in the steel [40]. The beginning and end temperature of the martensitic transformation of 95X18 steel with increased carbon content decreases when compared with the low carbon steel 20X13 as shown in Fig 4.2. Increased carbon content generally weakens the bonds between the neighbouring iron atoms and hence 95X18 steel with higher carbon content reaches higher plastic deflections during bending.

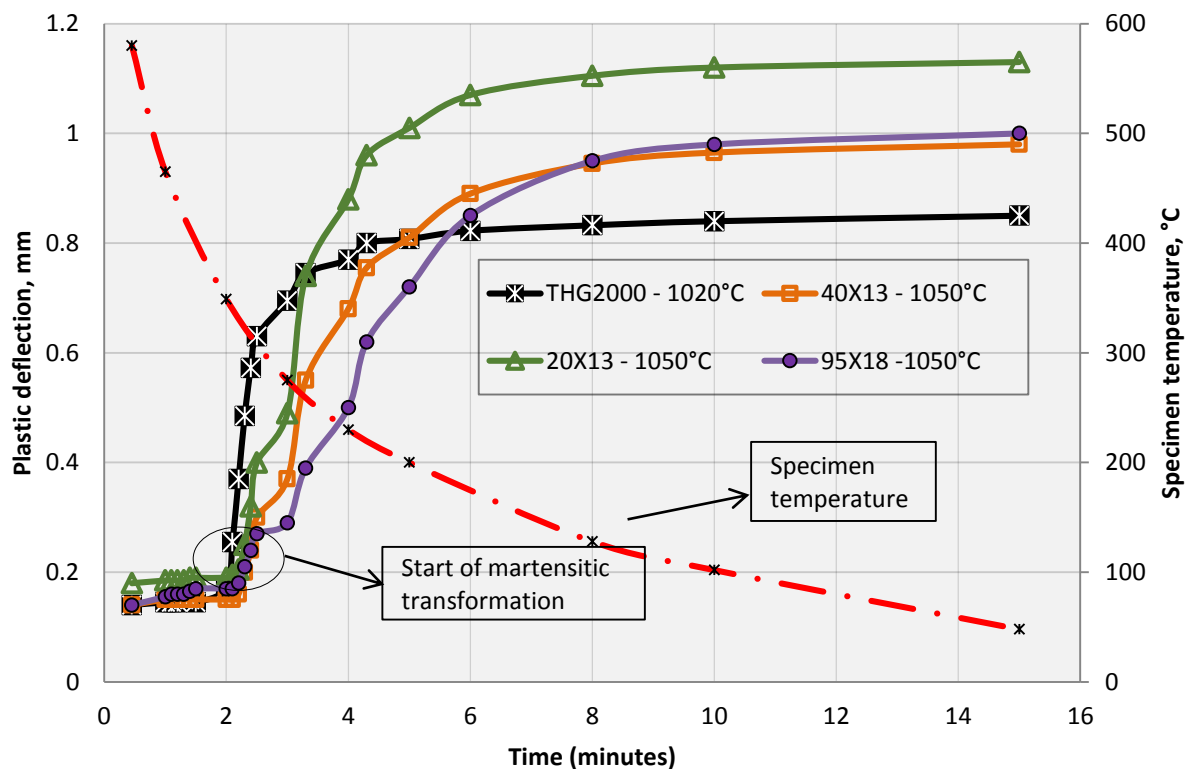


Fig. 4.2 Plastic deformation of different steel specimens air quenched from higher austenizing temperature 1020°C and 1050°C

THG2000 Steel with lower chromium content obtains plasticity transformation much earlier when compared with 95X18 steel with higher chromium content. Chromium carbides can effortlessly dissolve at temperature higher than 1000°C. It is also evident that 40X13, 95X18, 20X13 steels with higher chromium content possess higher plastic deflections than THG2000 steel at optimal hardening temperature. It may be due to the inhibitory influence of chromium on the segregation of carbon causes the difference in plasticity transformation [53].

After quenching, the steel has a microstructure consisting of martensite, retained austenite and carbides as shown in Fig. 4.3. Martensite is a forced solution of carbon in ferrite. The needle like martensite structures and the grain boundaries of an initial microstructure of ferrite are very clearly observed in Fig. 4.3. The martensite is extremely tough because of carbon atoms which become trapped during the very quick diffusion less phase transformation.

The amount of retained austenite increases with increasing alloying content, higher hardening temperature, longer soaking times and slower quenching [54]. Air hardening is engaged for steel with high hardenability, which occurs mainly due to the combined presence of manganese, chromium and molybdenum [49].

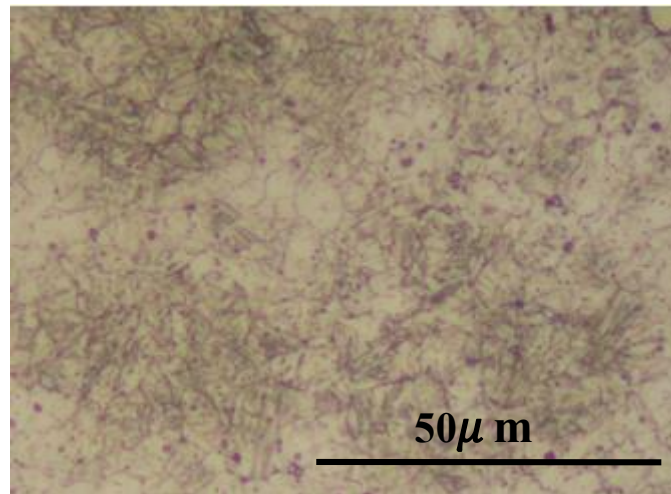


Fig. 4.3. Microstructure of THG2000 specimen air quenched from 1020°C

When quenching through the martensite range, which is certainly below the M_s temperature, the martensite which forms leads to an increase in volume and stresses in the material.

4.2. INVESTIGATION OF TRANSFORMATION PLASTICITY OF STEEL DURING TEMPERING

The steels mentioned at Table. 3.2 are investigated and tested in austenized, quenched and tempered conditions. The quenched specimens were tempered at 200°C to 600°C. THG2000 is a secondary hardening steel as the softening curves for these steels will show a range of tempering temperature in which the softening is arrested as it possess pretty high alloy content, which undergoes increase in the hardness joined with the precipitation of alloy carbides in tempered martensite at elevated tempering temperatures.

Yield strength increases with secondary hardening and plastic deflection drops to a minimum. Yield strength and bending strengths are at or close to their maximum values for tempering, after tempering at 500°C. Longer tempering times would shift the minimum in plastic deflection to lower tempering temperature [55].

During high temperature tempering, precipitated secondary (newly formed) carbides and newly formed martensite can increase the hardness. Characteristics of these phenomena are called secondary hardening. In Fig. 4.4 the steels 40X13, 95X18, 20X13 have a pronounced secondary hardening peak at 470°C - 480°C tempering temperatures, while for THG2000 the peak is shifted towards higher tempering temperature at 500°C.

It may be considered with different phenomenon. The first one is with relatively high alloying content like chromium the hardness is increased at 470°C - 480°C higher tempering temperature in case of steel 40X13, 95X18, 20X13 but softening of the material is found after 500°C as the material does not contain high molybdenum or vanadium content.

While in other case, during tempering of THG2000, elements like chromium, vanadium, and molybdenum precipitate with the carbon. As it contains fairly low concentrations of these elements, the softening of the steel can be retarded until much higher temperatures are reached, as they migrate to the carbide phase when diffusion is possible. This allows the steel to maintain its hardness in high temperature and also requires very high temperatures during tempering, to achieve a reduction in hardness. The molybdenum content retains the material hardness even at red hot temperature, while vanadium is mainly used to improve the toughness of the material [56, 57].

The hardened specimens were tempered at 1h at different temperature 200-600°C. Deflection and strength are enhanced to maximum value because residual stresses are relieved and transition carbides are precipitated in the martensite particularly at lower tempering temperatures, strength and ductility are subsequently decreased when carbides precipitates. It is clear that the diffusion of carbon need higher temperature as THG2000 is an alloyed steel. After the martensitic start temperature the diffusion of carbon takes place. At higher temperatures around 550°C the diffusion of chromium starts to takes place [51].

The austenizing temperature is one of the most important external factor which affects the hardness of the quenched and tempered steels at different tempering temperatures. From Fig. 4.4 it is highly apparent that 95X18 steel shows pronounced hardening peak for 1050°C when compared with 1000°C of austenizing temperature.

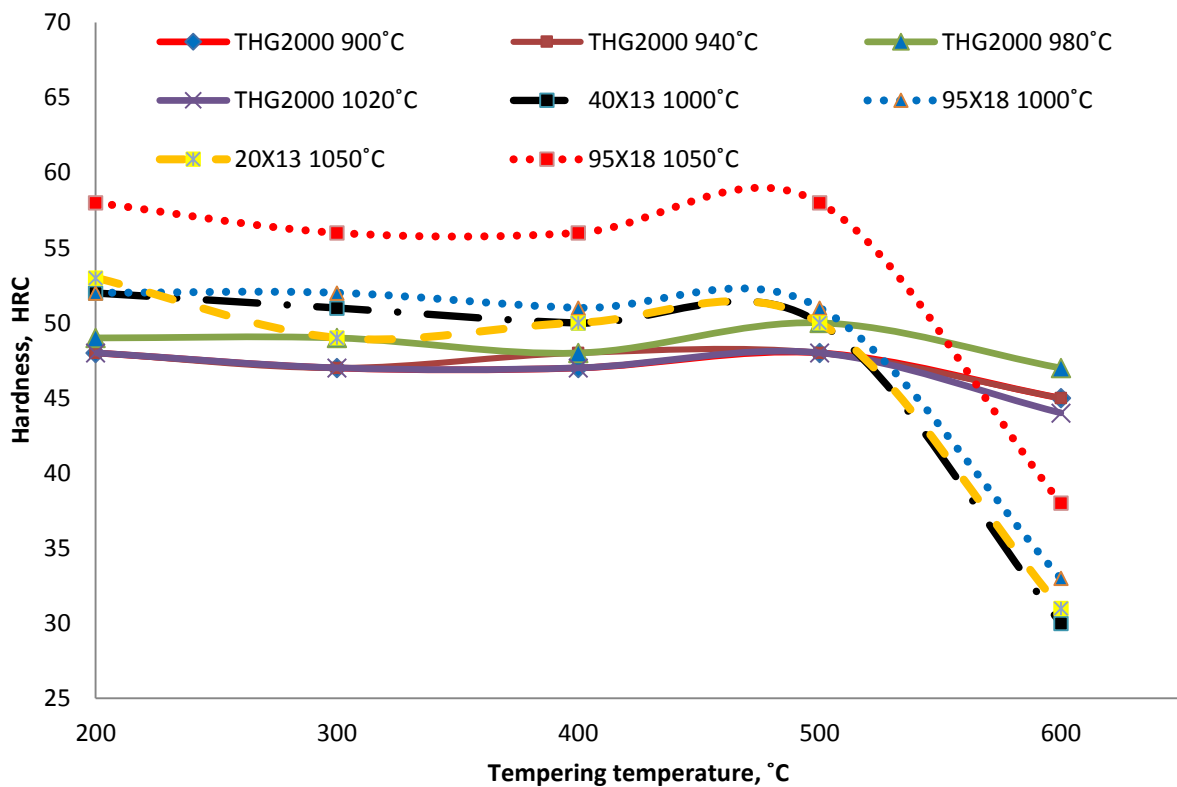


Fig. 4.4. Hardness of the tempered steels, austenized at temperatures (900°C to 1050°C), air quenched and tempered at different temperatures

The tempering temperature plays a vital role in deflection of the specimen due to applied stress of 100 MPa in a bending specimen. It should be noted that low temperature tempering only affects the martensite, but tempering at higher temperature also affects the retained austenite. The microstructure of high temperature tempering specimen consists of tempered martensite, newly formed martensite, some retained austenite and carbides. It is to be renowned that the austenizing temperature and chemical composition of the material also plays an efficient role in the deflection obtained.

The THG2000 specimens shows standard stabilized deflection values even at higher tempering temperatures as shown in Fig. 4.5 because of the presence of their alloying elements like molybdenum and vanadium. Whereas the higher chromium content in steels 40X13, 95X18, 20X13 with absence of molybdenum and vanadium alloying elements shows greater difference in their deflection rate especially at higher tempering temperature at 500°C. It may be due to the phenomenon that volume difference in tempered steel as during tempering, the volume decreases when carbon precipitate from martensite.

At temperature above 450°C the diffusion of chromium, carbon and iron proceeds whereas till 450°C the carbon atom segregates from solid solution. It explains the phenomenon that the hardened steel has temporal increased plasticity during tempering.

The low carbon chromium steel 20X13 during tempering shows greater deflection than the specimens with higher carbon content but they have stabilized deflection rate when compared with 95X18 steel shown in Fig. 4.5. This may be because in low carbon steel the tensile stress blocks the carbon precipitation from martensite at higher tempering temperature range of 500°C. Whereas 95X18 steel specimen starts bending backwards intensively after 500°C, at these temperature the intensive diffusion of C, Fe, Cr atoms occur with formation of special carbides generally in the dislocations [51], the precipitation of carbon goes with the diffusion of chromium atoms [58] and it may be also related to the auto deformation effect.

It is clear that austenizing temperature also plays an efficient role in deflection of specimens at different tempering temperature at a constant applied stress. The THG2000 steel specimen shows higher deflection curves at lower austenizing temperature of 900°C when compared with 980°C for different tempering temperatures and THG2000 steel possess stabilized deflection curve this is due to effect of vanadium and molybdenum alloying elements, while in 95X18 steel specimens the deflection values increases with increase in

austenizing temperature as it shows higher deflection values at 1050°C for different tempering temperatures.

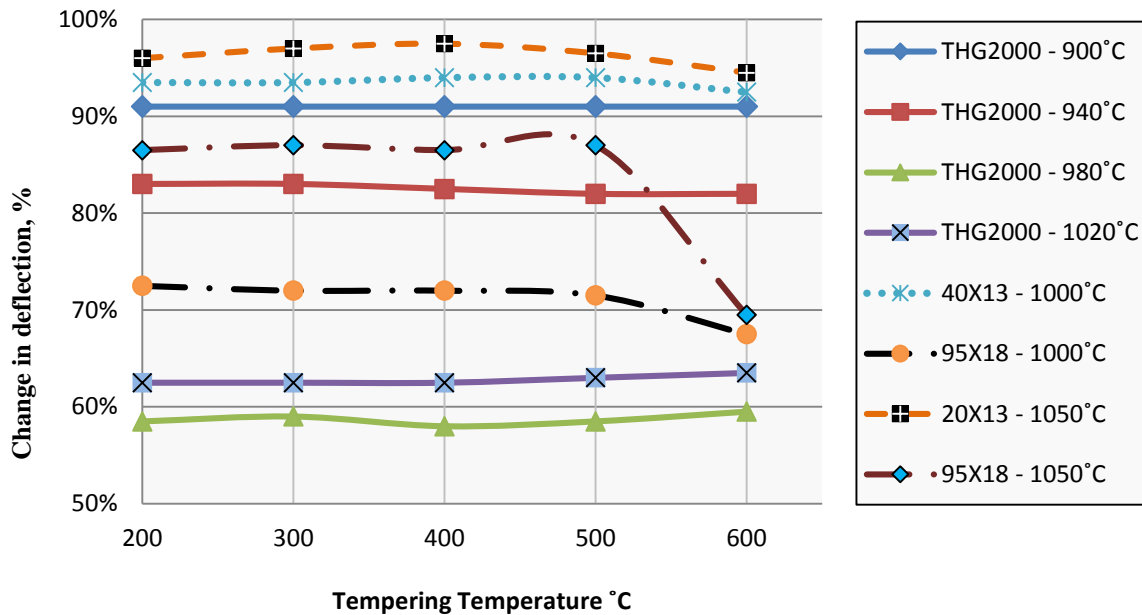


Fig. 4.5. Deflection of specimens austenized at temperatures (900°C to 1020°C), air quenched and tempered at different temperatures

4.3. EFFECT OF AUSTENIZING TEMPERATURES ON THE QUENCHED HARDNESS

In lower carbon content steel (20X13) noticeable quantities of ferrite may become stable at austenizing temperature. Fig. 4.6 shows the quenched hardness as a function of austenizing temperature in various steels with various levels of carbon demonstrating the effect of carbon and ferrite formation. Which explains the phenomenon on lowering the hardness of steels which possess lower carbon contents. Lower quenched hardness is followed by a lower tempered hardness as shown in Fig. 4.4.

At high austenizing temperature 1050°C, the quenched hardness is found effectively higher than the remaining austenizing temperatures. This effectively shows that austenizing

temperature directly proportional to the quenched hardness, and the higher carbon steel 95X18 possess the higher quenched hardness value at this temperature.

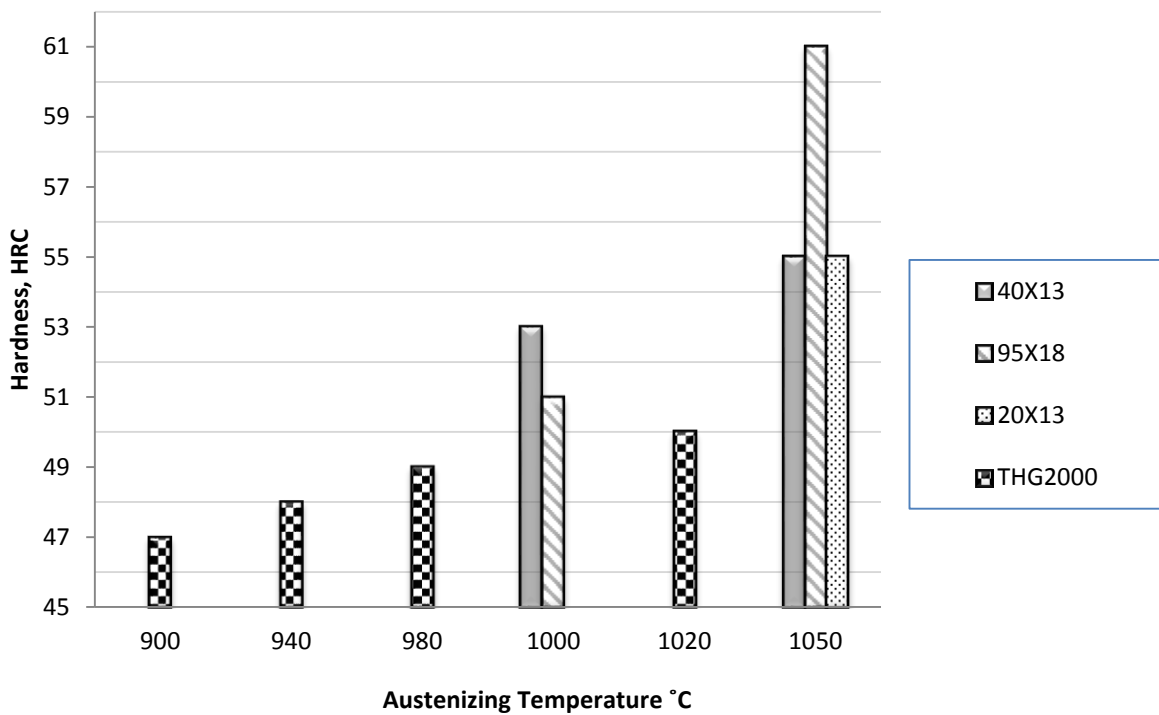


Fig. 4.6 Effect of austenizing temperature on the quenched hardness of different steels

The highest carbon steel is most sensitive to austenite formation at the highest austenizing temperature, and as a result has a lower quenched hardness than that of intermediate carbon content which is seen at 1000°C. Where the hardness of 95X18 steel with 0.97 wt% C found with lower quenched hardness compared with 40X13 steel with 0.35 wt% C content. However higher available carbon content in steel makes possible higher peak secondary hardness.

4.4. AUTODEFORMATION EFFECT DURING TEMPERING OF QUENCHED AND BENT SPECIMENS

The autodeformation effect is one of the interesting phenomenons found in plasticity transformation during tempering. It is named as autodeformation because the material deforms even the value of external stress acting is zero. This is mainly due to another martensitic transformation in the compressed and stretched part of the specimens. The stretched volume of specimens were increased which results in additional curving of bent and quenched specimens.

It is to be noted that during tempering the relative volume of retained austenite is lower when compared with the martensite. The influence of compression and tension stresses generated inside the specimens is different for the temperature at which the martensitic transformation starts [58]. The specimen is compressed due to the bending stress of 100 MPa acting on the specimen during bending test.

Tempering of the quenched and bent specimens were carried out which reveals the autodeformation effect related with increase in deflection around 2% due to the act of compression stresses and in some cases loss of deflection of specimens due to tension stresses as shown in Fig. 4.7. THG2000 quenched and bent specimen undergoes auto deformation effect at different tempering temperature. It is clearly identified that the specimen possesses loss in deflection at higher tempering temperature of 600°C.

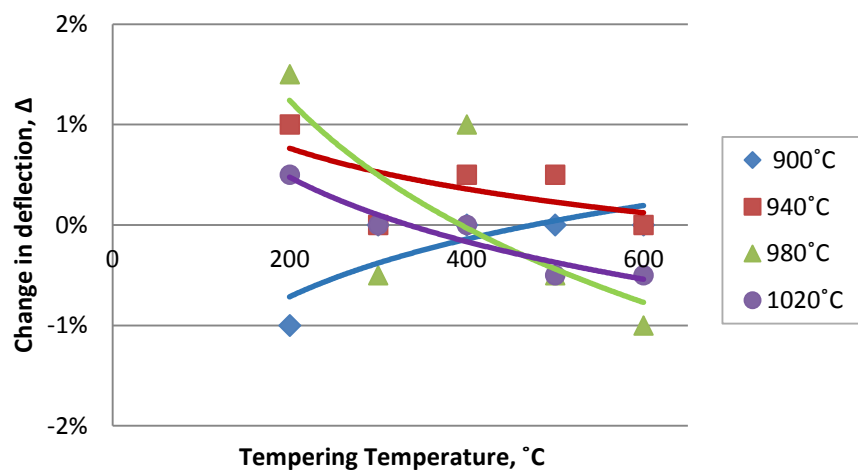


Fig. 4.7. THG2000 quenched and bent specimen having change in deflection at different tempering temperature

The auto deformation effect on different types of steels austenized different temperatures, quenched, bent and tempered is clearly explained in Fig. 4.8 and explains the fact that the specimen possesses more positive deflections, than loss in deflections. These steels show huge increase in deflection at higher tempering temperature of 600°C.

The stretched side of the specimen have less martensite compared to the compressed one during the moment of unloading as the decomposition of austenite into martensite in the compressed part of the specimen must be taking place in advance [59].

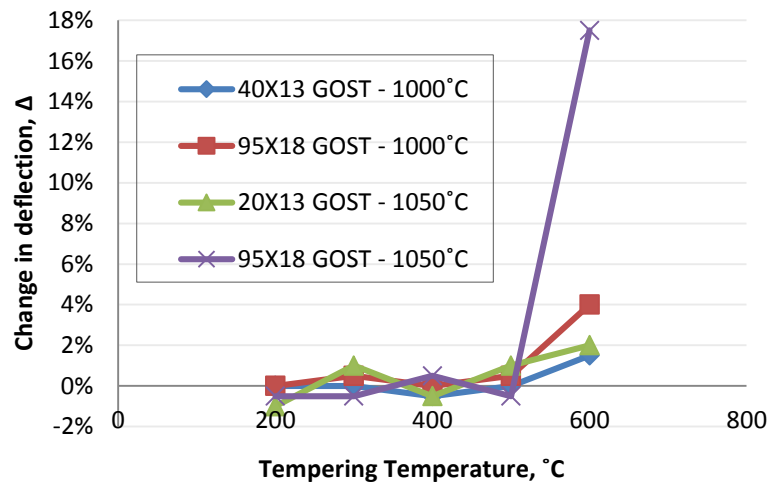


Fig. 4.8. Different quenched and bent steel specimen having change in deflection at different temperig temperature

The 95X18 steel posses peak deflection increament of about 18% at 1050°C austenizing temperature, which clearly indicates the fact the austenizing temperature plays an unique role in transformation expansion. This may be due to the phenomenon that increasing the austenizing temperature, more and more carbides are dissolved in austenite and the increased carbon content weekens the blinds between neighbouring iron atoms and thus the specimens reach higher plastic deflections.

5. CONCLUSION

- The transformation plasticity of THG2000 specimen increases 25% to 35% when austenizing temperature decreases from 1020°C to 900°C as austenite enriches with C and Cr which happens mainly because chromium carbides effortlessly solves in matrix at higher austenizing temperature. THG2000 steel possess higher M_s (370°C) at lower austenizing temperature (940°C) and decreases to 310°C (M_s) when austenizing temperature (1020°C) is increased.
- Plasticity transformation of the specimen is directly proportional to the austenizing temperature of the quenched specimen as in 1020°C the plasticity transformation starts at 4 minutes whereas in 900°C it starts prior around 3 minutes.
- The martensitic start temperature (M_s) directly depends on the amount of carbon content and austenizing temperature. M_s is found lower for 95X18 steel with higher carbon content when compared with 20X13 low carbon steel.
- THG2000 Steel with lower Cr content obtains plasticity transformation much earlier when compared with 95X18 steel with higher Cr content. Chromium carbides can effortlessly dissolves at temperature higher than 1000°C. 40X13, 95X18, 20X13 steels with higher Cr content possess higher plastic deflections than THG2000 steel at optimal hardening temperature due to the inhibitory influence of Cr on the segregation of carbon.
- The steels 40X13, 95X18, 20X13 have a pronounced secondary hardening peak at 470°C - 480°C tempering temperatures but softening of the material is found after 500°C due to the intensive diffusion of C, Fe, Cr atoms occur with formation of special carbides as the material does not contain high molybdenum or vanadium content. While for THG2000 the peak is shifted towards higher tempering temperature at 500°C and stabilized peak was obtained.
- The quenched and bent specimen undergoes auto deformation effect around 10% deflections at different tempering temperature due to the act of compression and tensile stresses. The stretched volume of specimens were increased which results in further curving of bent and quenched specimens. The material deforms due to another martensitic transformation in the compressed and stretched part of the specimens.

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