The Investigation on Strain Strengthening Induced Martensitic Phase Transformation of Austenitic Stainless Steel: A Fundamental Research for the Quality Evaluation of Strain Strengthened Pressure Vessel

To cite this article: Bo Li et al 2018 IOP Conf. Ser.: Earth Environ. Sci. 128 012005

View the article online for updates and enhancements.

Related content
- The Effect of Strain Hardening on Mechanical Properties of S30408 Austenitic Stainless Steel: A Fundamental Research for the Quality Evaluation of Strain Strengthened Pressure Vessel Bo Li, Fa Cai Ren and Xiao Ying Tang
- Mechanical Behaviour of 304 Austenitic Stainless Steel Processed by Room Temperature Rolling Rahul Singh, Sunkulp Goel, Raviraj Verma et al.
- Hydrogen Environment Embrittlement on Austenitic Stainless Steels from Room Temperature to Low Temperatures Toshio Ogata
The Investigation on Strain Strengthening Induced Martensitic Phase Transformation of Austenitic Stainless Steel: A Fundamental Research for the Quality Evaluation of Strain Strengthened Pressure Vessel

Bo LI, Fa Cai REN, Xiao Ying TANG
Shanghai Institute of Special Equipment Inspection and Technical Research (SSEI), Shanghai 200333, China
Email: libo@ssei.cn

Abstract: The manufacture of pressure vessels with austenitic stainless steel strain strengthening technology has become an important technical means for the lightweight of cryogenic pressure vessels. In the process of increasing the strength of austenitic stainless steel, strain can induce the martensitic phase transformation in austenite phase. There is a quantitative relationship between the transformation quantity of martensitic phase and the basic mechanical properties. Then, the martensitic phase variables can be obtained by means of detection, and the mechanical properties and safety performance are evaluated and calculated. Based on this, the quantitative relationship between strain hardening and deformation induced martensitic phase content is studied in this paper, and the mechanism of deformation induced martensitic transformation of austenitic stainless steel is detailed.

1. Introduction
Austenitic stainless steel has been widely used in energy, chemical, aerospace and other fields. In recent years, the manufacture of pressure vessels with austenitic stainless steel strain strengthening technology has become an important technical means for the lightweight of cryogenic pressure vessels. The key technology and safety evaluation method related to the strain strengthened stainless steel cryogenic container need to be studied [1-3]. In the process of increasing the austenitic stainless steel vessel strength, strain can induce the martensitic phase transition in austenite phase. There is a quantitative relationship between the transformation quantity of martensitic phase and the basic mechanical properties. The increase of martensite content within a certain range can play a gain in the resistance to damage and fatigue resistance of the material. However, the excessive martensitic transformation is harmful to the ductility and plasticity of the material, thus increasing the risk of brittle failure at low temperature [3-5]. In the process of manufacturing thin-walled pressure vessels by strain hardening process, if the deformation control is not precise, it is easy to cause excessive plastic deformation or uneven deformation of the whole container structure [5]. Therefore, it is very necessary to evaluate the nondestructive safety of the strain strengthened pressure vessel and its key force. There is a quantitative relationship between the transformation quantity of martensitic phase and the basic mechanical properties. The change of toughness and plasticity is directly related to strain hardening for austenitic stainless steel. Then, the martensitic phase variables can be obtained by means of detection, and the mechanical properties and safety performance are evaluated and calculated. Based on this, the quantitative relationship between strain intensification and the content of martensite phase induced by
deformation is established in this paper. The mechanism of martensitic transformation induced by deformation of austenitic stainless steel is also studied.

2. Experimental Details

2.1. Preparation of Strain Strengthened Stainless Steel Specimens

The 8mm and 10mm thickness parent plates of UNS-S30408 austenitic stainless steel are used in the strain strengthening experiment. The plates are prepared as standard specimens for strain strengthening via mechanical tensile method. The strain rate of 0.5mm/min is tailored to strengthen the mechanical tensile strain specimens with different thickness. In the strain hardening process, the strain is precisely controlled and measured with the aid of an extensometer installed on the specimen of a tensile test machine. Strain strengthening of mechanical stretching is set to seven groups according the strain stretch length, that is, 4%, 6%, 8%, 10%, 12%, 14%, and 16% of the tensile part of specimens. The specimens after mechanical tensile pre treatments are shown in Fig.1.

2.2. Observation and Analysis on the Characteristics of Strain Induced Martensitic Transformation

The longitudinal section of plate rolling direction were selected to observe the metallographic analysis, including microstructure, residual austenite ferrite, martensite. In the pretension test, the extensometer is used to accurately control the strain. For the partially reinforced tensile specimens, small sample pieces are intercepted at the center due to the maximum deformation at the center of the standard distance. The material phase analysis was carried out by X ray diffraction (XRD). The sample was scanned by Co target X ray diffractometer at 40° -85°. The step length of XRD is 0.02°. According to the data collected by the diffractometer, the diffraction peaks are determined. In addition, the integral strength of the distance between the corresponding plane and the diffraction peak is calculated. The volume fraction of each phase can be calculated according to the integral intensity of all diffraction peaks of each phase, which is proportional to the volume fraction of corresponding phase in the sample.

\[ I_i^{hkl} = \frac{KR_i^{hkl}V_i}{2\mu} \]

Figure 1 The Size Diagram of S30408 Specimen Size for Strain Strengthening Treatment (Unit: mm)

The quantitative estimation of phases by X-ray diffraction is based on the principle that the total integrated intensity of all diffraction peaks for each phase in a mixture is proportional to the volume fraction of that phase. If the grains of each phase are randomly oriented, the integrated intensity ‘I’ of any diffraction peak from phase ‘i’ is given by [6]

where,

- \( I_i^{hkl} \): integrated intensity for (hkl) plane of i-phase;
- \( K \): the instrument factor;
- \( R_i^{hkl} \): material scattering factor and depends on X ray diffraction angle, interplanar spacing of [hkl], composition and the crystal structure of the phase i;
$V_i$: volume fraction of phase $i$; 
$\mu$: linear absorption coefficient.

As martensite has ferromagnetism, the ferromagnetic measuring instrument can be used to detect the martensitic phase variables of the strain strengthened austenitic stainless steel. Ferromagnetic content analyzer is also used to detect ferrite content in austenitic stainless steel welds. The method is nondestructive testing, less time-consuming, and can be detected on line in real time. In addition, during the actual operation, when the martensite content is less than 2%, the ferromagnetic tester is often unable to detect the results. Therefore, it is necessary to modify the data of martensitic ferromagnetic detection by XRD quantitative analysis.

3. Evolution Mechanism of Martensitic Transformation by Strain Strengthening

The metastable austenitic stainless steel produces martensitic transformation when it is deformed. The metastable austenitic stainless steel produces deformable martensitic transformation when the material is subjected to tensile or fatigue testing. The order of phase transition is austenite phase $\gamma \rightarrow$ martensite phase $\varepsilon \rightarrow$ martensite phase $\alpha'$ or martensitic phase $\alpha'$ directly from austenite phase $\gamma$. The $\gamma$ phase is a face centered cubic structure (FCC) with low strength and good toughness and plasticity. The $\varepsilon$ phase is close packed structure (HCP), the six party is less, by the middle of the austenite phase $\gamma$ to $\alpha'$ martensite phase transformation. The $\varepsilon$ phase gradually disappeared along the process of strain.

The influence of strain rate on the martensitic transformation is mainly related to the plastic deformation mechanism and the deformation thermal effect. In the normal temperature stretching process, the effect of thermal effect on deformation is negligible when the tensile velocity is slow (the strain rate 0.5mm/min).

Rapid tensile specimen (strain rate 2mm/min, 5mm/min, 8mm/min), in a small amount of deformation (less than 16%), the plastic deformation mechanism of the speed control (such as cross slip and grain boundary sliding etc.) can not be induced, resulting in the local stress concentration at grain boundaries. A large number of defects appeared in the shear band. Martensite promotes the transformation of martensite in the intersection point of the shear zone with large deformation defects. The martensite, which grows at the intersection point of the shear zone, forms a large grid frame and irregular strip. Martensite is distributed between the austenite and the austenite. So the amount of martensite produced by the rapid tensile specimen is more than that of the slow tensile specimen.

However, it is necessary to point out that if the deformation is too large, the effect of thermal effect on deformation should be considered. The thermal effect of the temperature rise increases the stacking fault energy of austenitic stainless steel, reduces the thermodynamic driving force of martensitic transformation, and increases the mechanical driving force required for the phase transition. Therefore, although the high strain rate can produce more shear bands, it can not reach the driving force of the phase change. The velocity of martensite transformation slows down, and the amount of transformation is difficult to increase significantly.

The strain is the key parameter to determine the yield strength of the material. If the strain is too small, the material is not strengthened enough, the material saving effect and economic benefits are not outstanding. Excessive strain will cause excessive loss of plasticity and toughness of materials, and affect the safe operation of pressure vessels. Although the current relevant documents and standards require the maximum control of austenitic stainless steel, the maximum strength of the strain is not more than 10%. But in order to further tap potential and analysis of large strain, strain dependent on material properties, this experiment will be set in the range of variables is less than or equal to 16%.

The experimental results show that when the strain rate is fixed, the martensitic phase ratio increases with the increase of strain deformation. After the austenite grain is dynamically recrystallized, the grain size is further refined, and the average grain size decreases with the increase of the deformation amount.

The microstructure observation shows that thicker plate-like $\alpha'$ martensite and fine-grained needle $\alpha'$ martensite tend to form parallel strips in the same austenite grain, forming parallel or cross...
relationship between strip and bundles, as shown below.

![Figure 2 The Micro-structure Characteristics of Martensite](image)

4. The Influence of Different Strain Degrees on Martensitic Transformation

The XRD diffraction peaks of 8mm and 10mm plate specimens are shown as follows. The volume percentage of austenite and martensite can be calculated in Tab.1 and Tab.2.

| Specimen (Pre-tensile Strain Rate of 0.5mm/min) | Calculation of the Volume Percentage of Martensite Phase | Measurement of Volume Percentage of Martensite Phase |
|-----------------------------------------------|----------------------------------------------------------|-----------------------------------------------------|
| 8mm Thickness Plate with pre-strain length of 4% | 0.3825%                                                   | (undetected)                                        |
| 8mm Thickness Plate with pre-strain length of 6% | 0.9366%                                                   | (undetected)                                        |
| 8mm Thickness Plate with pre-strain length of 8% | 2.0455%                                                   | 3.0%                                                |
| 8mm Thickness Plate with pre-strain length of 10% | 4.7182%                                                   | 4.7%                                                |
| 8mm Thickness Plate with pre-strain length of 12% | 6.4720%                                                   | 5.2%                                                |
| 8mm Thickness Plate with pre-strain length of 14% | 9.0417%                                                   | 8.1%                                                |
| 8mm Thickness Plate with pre-strain length of 16% | 10.7225%                                                  | 10.1%                                               |
| 10mm Thickness Plate with pre-strain length of 4% | 3.9038%                                                   | 4.9%                                                |
| 10mm Thickness Plate with pre-strain length of 6% | 5.5521%                                                   | 5.5%                                                |
| 10mm Thickness Plate with pre-strain length of 8% | 5.3819%                                                   | 5.7%                                                |
| 10mm Thickness Plate with pre-strain length of 10% | 0.7430%                                                   | (undetected)                                        |
| 10mm Thickness Plate with pre-strain length of 12% | 1.1216%                                                   | 2.1%                                                |
| 10mm Thickness Plate with pre-strain length of 14% | 3.8235%                                                   | 3.8%                                                |
| 8mm Thickness Plate with pre-strain length of 16% | 4.2150%                                                   | 4.5%                                                |

Based on the calculated values, it can be basically predicted that when the strain rate is less than 16%, when the strain rate is 0.5mm/min, the percentage of martensitic volume fraction will increase gradually. The maximum value of the total is about 10%. However, it is also necessary to explain that the numerical results based on the XRD calculation still have an inevitable deviation from the actual situation. The main reason is: ignoring the influence of phase composition outside of austenite and martensite material; influence of XRD diffraction parameters and diffraction peak deviation; I value of...
computer processing for low angle fracture tip may exist fuzzy processing; high angle peak calculation, there may be a phase of peak drift even with similar material peak overlap and so on. In addition, in the field detection section of engineering, it is difficult to detect XRD by destructive sampling of multiple fixed points for strain intensified pressure vessel equipment. It is still necessary to determine the proportion of martensitic phase by means of nondestructive testing. The calculated value of XRD in this experiment can be used to compare the experimental results with the results of NDT.

The average value of ferromagnetic martensitic phase volume percentage measured for pre stretched specimens is obvious. With the increase of pre strain, the percentage of martensitic volume increases almost linearly, and the maximum value is about 12%. After comparing the calculated data of volume fraction of martensitic volume with XRD data after pre stretching, it can be seen that the calculated percentage of martensitic volume percentage of XRD data of 10mm specimen after pre stretching is much higher than that of ferromagnetic martensitic volume percentage measurement. Although the thickness of the 8mm plate is two, there is a certain degree of deviation. But the basic trends are consistent. This shows that, at room temperature is less than 16% of the pre tension should be variable, with pre tension strain, deformation induced martensite phase showed increasing trend linear variable. The test results of ferromagnetic tester can basically reflect the deformation induced martensitic phase ratio of more than 6% of the strain.

![Figure 3](image-url)  
**Figure 3** XRD Diffraction Peak of 8mm Thickness Plates with Different Pre-strain Lengths (The Percent of Parent Material Specimen for Mechanical Tensile): (a) 0%; (b) 4%; (c) 6%; (d) 8%; (e) 10%; (f) 12%; (g) 14%; (h) 16%.
5. Conclusion

This paper researched and established the quantitative relationship between strain hardening of austenitic stainless steel S30408 and the degree of deformation induced martensite phase content. The experiment selected deformation length parameters of 4%, 6%, 8%, 10%, 12%, 14%, 16%, two kinds of austenitic stainless steel plates with different thickness, the pre tensile strain hardening. In this paper, the mechanism and characteristics of martensitic transformation induced by strain hardening are studied, and the specific phase analysis of austenitic stainless steel material based on XRD corresponding to transformation is carried out. With the increase of pretension stress, the volume percentage of martensitic phase increases almost linearly, and the maximum value is about 12%. After comparing the calculated data of volume fraction of martensitic volume with XRD data after pre stretching, it can be seen that the calculated percentage of martensitic volume percentage of XRD data coincides with the average value of ferromagnetic martensitic volume percentage measurement. The experimental results are helpful for obtaining martensitic variables of strain strengthened austenitic stainless steel pressure vessel detection parts by means of detection, and evaluating and calculating their mechanical and safety performance.

Acknowledgments

The work was sponsored by Shanghai Bureau of Quality and Technical Supervision Research Project (Grant No.2015-38) and the National Natural Science Foundation of China (Grant No.51505293).

References

[1] Garner F A, Greenwood L R, Harrod D L. Potential high fluence response of pressure vessel internals constructed from austenitic stainless steels[J]. 2013.
[2] Auzoux Q, Allais L, et al. Effect of pre-strain on creep of three AISI 316 austenitic stainless steels in relation to reheat cracking of weld-affected zones[J]. Journal of Nuclear Materials, 2010, 400(2):127-137.
[3] Chen S P, Rui W U, Tan F G. Finite Element Analysis of Opening Reinforcement for Strain Strengthening Pressure Vessel[J]. Petro-Chemical Equipment, 2013, 35(4):23-32.
[4] Zhao Z. Strain Hardening of Austenitic Stainless Steel Pressure Vessels[J]. Modern Manufacturing Technology & Equipment, 2016.
[5] Chen T, Wang B M, Tao X U, et al. Research Status of Strain-Strengthening Technology for Austenitic Stainless Steel Pressure Vessels and Comparison of Foreign Standards[J]. Materials for Mechanical Engineering, 2012, 36(3):1-4.
[6] De A K, Murdock D C, Mataya M C, et al. Quantitative measurement of deformation-induced martensite in 304 stainless steel by X-ray diffraction[J]. Scripta Materialia, 2004, 50(12):1445-1449.