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Two Uncoating Techniques for Measuring Cold-Formed Steel Residual Stress Using Cos-α X-ray Diffraction Method

Tri Widya Swastika [a,*], Heru Purnomo [a], Henki Wibowo Ashadi [a], Muhammad Refai Muslih [b], Rifky Apriansyah [b], Mohd Syahrul Hisyam bin Mohd Sani [c]

[a] *Civil Engineering Department, Universitas Indonesia, Jl. Lingkar, Depok, Indonesia*
[b] *Neutron Scattering Lab. PRTDRAN-BRIN, Serpong, Indonesia*
[c] *Civil Engineering Studies, Collage of Engineering, Universiti Teknologi MARA, Pahang, Malaysia*
Corresponding author: [*]tri.widya@ui.ac.id

Abstract*—The cold-bending effect during the roll-forming process may affect the material's mechanical properties and induce residual stress in the cold-formed steel sections. Cos-α X-ray Diffraction is an appropriate method for measuring residual stress in cold-formed steel due to the materials' thinness. This method also offers excellent precision and simplicity. However, the limited penetrating ability of X-rays, which extend only a few microns, significantly hinders the measurement of residual stresses in cold-formed steel when coatings are present. Therefore, this study will implement two uncoating or de-coating techniques for measuring residual stress using the cos-α X-ray Diffraction method on the surface of cold-formed steel with a 50 µm layer of aluminum-zinc coating. These techniques include water sanding and chemical solutions. Two procedures are performed for the chemical solution: the first procedure combines a 25% hydrochloric acid (HCl) solution with a 25% ammonium hydroxide (NH_4OH) solution, while the second procedure uses only a 25% hydrochloric acid (HCl) solution. This study demonstrates that the second procedure effectively removes the surface coating from cold-formed steel and provides a good classification of cos-α X-ray Diffraction intensity data related to the Debye-Scherrer ring. A combination of 25% hydrochloric acid (HCl) and 25% ammonium hydroxide (NH_4OH) solution results in a mediocre classification. On the other hand, the water sanding technique produced more bad classifications. Furthermore, the key to the success of the cos-α X-ray Diffraction method is removing the coating from the cold-formed steel.

***Keywords*—Uncoating techniques; cold-formed steel; residual stress; Cos-α X-ray diffraction method.**

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I. Introduction

Cold-formed steel sections are generally manufactured by roll-forming and press brake operation [1], [2], [3]. People commonly use roll-forming to increase production capacity [4]. The manufacturing of cold-formed steel through roll-forming serves as a concluding procedure before the profiles enter the construction sector. Several manufacturing processes precede this final procedure. Cold Rolled Coil (CRC) could be the starting point for the middle process. A cold rolling process in a cold rolling mill facility produces CRC, a steel product in the form of a coiled steel sheet. The process is continued with annealing, a heat treatment application that follows a specific temperature curve to satisfy specific mechanical properties requirements. Applications for frame housing and general structures require the CRC to proceed to the next step of applying protective coating. Some of these protections are coated, galvanized, or galvalume coated. The process of coiling and uncoiling takes place in between the stages mentioned [5]. Figure 1 displays the roll-forming machine, while Figure 2 illustrates the roll-forming process for creating cold-formed steel.

Fig. 1 Roll-forming machine

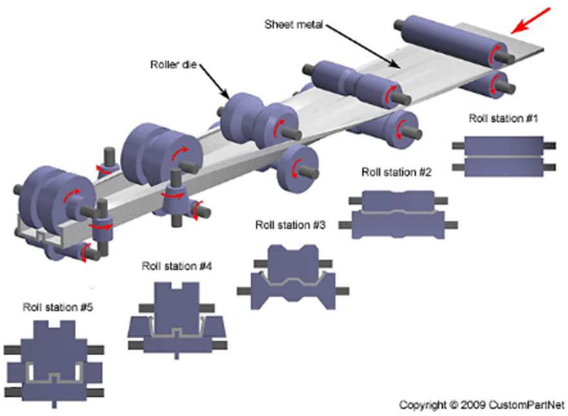


Fig. 2 Roll-forming process [5]

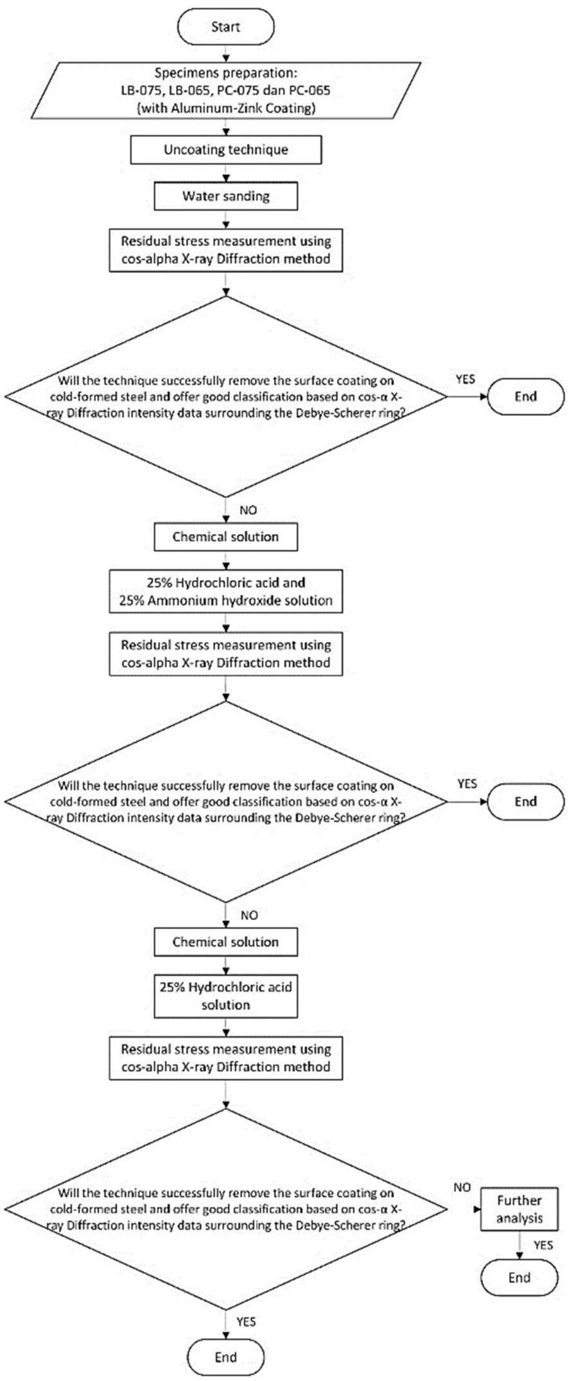


Fig. 3 Flow chart study

Due to the cold-bending effect during the roll-forming process, it can modify the mechanical properties of the material and induce residual stress in the cold-formed steel [4], [6]-[17]. With the potential to exceed 50% of the material's yield stress [9], [18]. residual stress has a significant impact on the design of cold-formed steel sections and their structural performance [10], [14]. Residual stress also influences the behavior, and ultimate strength of cold-formed steel members and causes a reduction in load-carrying capacity [16], [17], [19], [20], [21]

Typically, three types of methods are used to measure residual stress in steel: non-destructive, semi-destructive, or destructive [22]-[30]. One of the measurement methods that is commonly used in non-destructive tests that directly measure the steel is the X-ray Diffraction (XRD) method. The fundamental principle of X-ray stress measurement relies on the diffraction of X-rays by crystalline structures [15], [31]. Lester and Aborn introduced this method in 1925, and X-ray diffraction began to compete with mechanical methods after 1930. Bragg's law establishes the essential principles of X-ray diffraction analysis [13]. The X-ray Diffraction method reveals elastic stresses according to Brag's law by detecting changes in the lengths of crystalline planes in the microstructures of materials [32]. Several studies assert the high accuracy and simplicity of the X-ray Diffraction method [18], [29], [31], [33]- [36].

Cos-α X-ray Diffraction is an appropriate method for measuring residual stress in cold-formed steel due to the materials' thinness. In the previous studies by [37], [38] used the cos-α X-ray Diffraction method to measure the residual stress on cold-formed steel sections. The specimen's surface was uncoated using sandpaper (water sanding) [37], and a chemical solution [38]. They found that the Debye-Scherer ring can read better from the uncoating surface than from the coating surface.

Furthermore, Abvabi [12] reveals that assessing residual stress throughout a thin specimen's thickness typically involves removing material layers by electropolishing. He employed the XRD method in conjunction with electropolishing for removing layers of 50 μm [12], [30]. The ASTM E1558-09 Standard [39] delineates a recommended procedure for electropolishing while preventing the introduction of supplementary residual stresses.

However, the limited penetrating ability of X-rays, extending only a few microns, significantly hinders the measurement of residual stresses in cold-formed steel when coatings are present. This study implements two uncoating techniques for measuring residual stress using the cos-α X-ray Diffraction method on the surface of cold-formed steel with an aluminum zinc coating. The chemical solution undergoes two procedures: the first combines a 25% hydrochloric acid (HCl) with a 25% ammonium hydroxide (NH_4OH) solution, while the second procedure utilizes a 25% hydrochloric acid (HCl) solution exclusively.

II. Materials and Method

The materials and methods used in this study include preparing the specimen, using uncoating techniques, measuring residual stress using cos-α cos-X-ray diffraction, and analyzing the results, as illustrated in the flow chart in Figure 3.

A. Specimens Preparation

Cold-formed steels with a yield stress of 550 MPa and a coating of AZ100 from two different local manufacturers were used to make the specimens. These specimens have a 50 µm layer of aluminum zinc on top of them. Figure 4 shows the dimensions of the specimens, which have thicknesses of 0,60 and 0,70 mm (BMT), with LB on the left and PC on the right. Figure 5 illustrates the details of AZ100 B. In the specification, the letters denote the type of coating, while the number represents the mass in grams per square meter on both sides. AZ100 indicates an aluminum zinc coating of 100 grams per m².

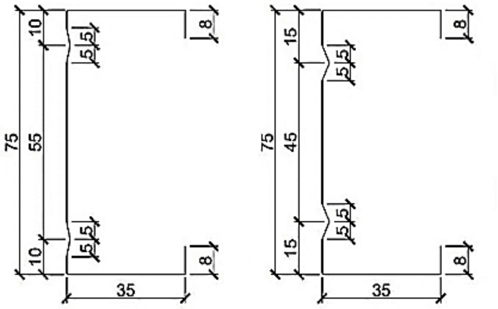


Fig. 4 Specimens dimension, LB (left) and PC (right) [37]

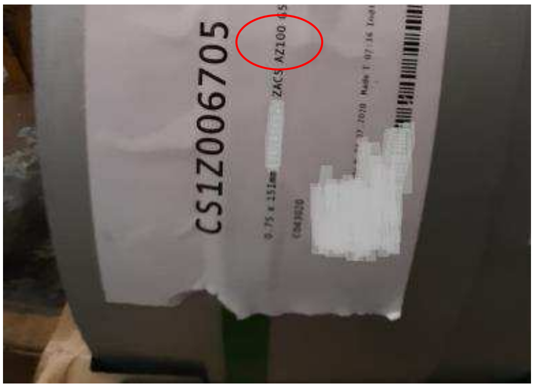


Fig. 5 Information on AZ100

B. Experimental Techniques

Two uncoating techniques were used to measure residual stress in cold-formed materials using cos-α X-ray Diffraction conducted at PRTDRAN-BRIN, Serpong, Indonesia.

1) Water Sanding Technique:

The first uncoating technique for measuring residual stress in cold-formed steel using the cos-α X-ray Diffraction method is water sanding. This technique is conducted manually. During the sanding procedure, utilize water (water sanding) and apply sandpaper grit ranging from coarse to fine, specifically P2000, P1500, P1000, P800, P600, P400, P320, P220, and P120, as seen in Figure 6, while the following Figure 7 displays the results.

Fig. 6 Variations of sandpaper grit for water sanding.

Fig. 7 Cold-formed steel specimens with coating and uncoating; a lip channel C-section (left) and flat steel sheet (right)

2) Chemical Solution Technique:

The second technique for measuring residual stress in cold-formed steel using the cos -α X-ray diffraction method is the chemical solution method. The chemical solution undergoes two procedures: the first combines a 25% hydrochloric acid (HCl) with a 25% ammonium hydroxide (NH_4OH) solution, while the second procedure solely uses a 25% hydrochloric acid (HCl) solution. This study uses a chemical solution, as illustrated in Figure 8. The process, depicted in Figure 9, occurs in a glass-lined room equipped with automatic ventilation. Applying this technique requires the use of rubber gloves and a mask.

Fig. 8 Chemical solution of 25% hydrochloric acid (HCl) and a 25% ammonium hydroxide (NH4OH) solution

Fig. 9 Preparing materials at BRIN Laboratories, Serpong

The first procedure uses two specimens: a steel plate and a cold-formed steel lip channel C-section. For a steel plate, cut the specimens to a 3 x 3 cm size on the steel plate. After that, weigh, give a label, and pour the chemical solution into the breaker glass, as seen in Figure 10. Apply a 1:1 ratio of 25% hydrochloric acid (HCl) with a 25% ammonium hydroxide (NH_4OH), using 300 ml of each solution. To operate, press the start button on the mixing machine simultaneously with the clock or alarm. The driving machine operates at a low speed. After some time, the chemical solution turns black, indicating that the coating on the specimen has dissolved. Then, turn off the machine and record the time.

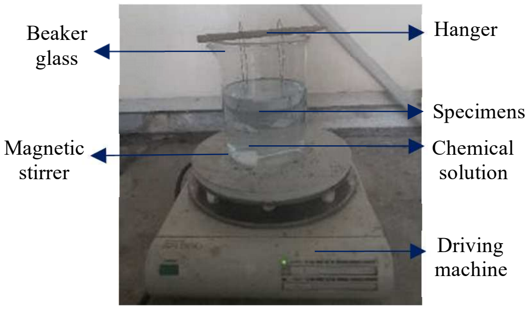


Fig. 10 The driving machine set up for the steel plate

The cold-formed steel lip channel C-section was treated with a solution equal parts 25% hydrochloric acid (HCl) and 25% ammonium hydroxide (NH_4OH). 1250 ml of each solution was used. Prepare the stirrer and mixture. Then, using a hanger, dip a portion of the specimen into the chemical while the driving machine is at average speed. A few hours should pass before the covering starts to peel off, as shown in Figure 11.


Fig. 11 The driving machine set up for the cold-formed steel lip channel C-section

For the second chemical solution technique procedure, a 25% hydrochloric acid (HCl) solution is utilized exclusively. To implement this procedure, we must remove the specimen while wearing rubber gloves. After that, wash the specimen with running water, wipe it, and place it in a desiccator tube for several minutes until it dries completely. Then, spray it with rust and corrosion protection; also, weigh the uncoated specimens. Finally, use the cos-α X-ray Diffraction apparatus to measure the residual stress on the uncoated specimen's surface. Figure 12 serves as the stage for explaining the uncoating techniques used in this second procedure.

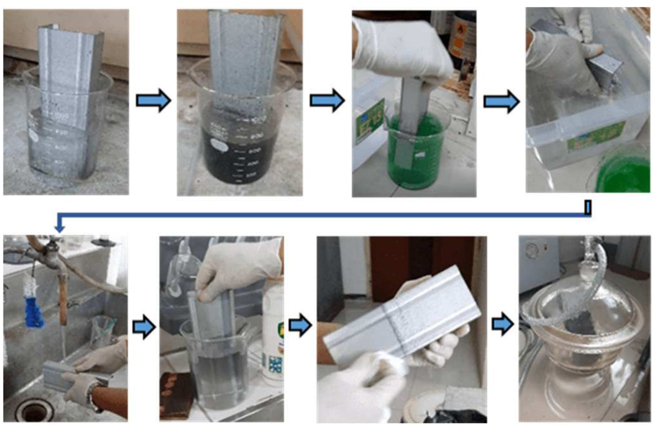


Fig. 12 Specimen dipping process

C. The Cos-α X-ray Diffraction Method

In the previous study [37], [38] used the cos-α X-ray Diffraction method on a specimen that was less than 1 mm thick to determine the residual stress on the surface in the work region. This is also known as plane stress. This technology can be applied in very thin specimens (penetration depth is about100 µm – 17 mm) and gives excellent accuracy, around 20 MPa [29]. If the material specimen under test satisfies the following requirements: it must possess a crystal structure, contain tiny grains, and have a known elastic constant, then the X-ray diffraction measurement is appropriate for residual stress analysis.

Cos-α X-ray SmartSite RS portable equipment from Rigaku has performed diffraction measurements. The X-ray tube uses a chromium corresponding to a diffraction angle, 2θ, of approximately 156°. The cos-α method was initially introduced by Taira, Tanaka, and Yamasaki in 1978 to analyze in-plane biaxial stress. The method employs the whole Debye-Scherrer ring captured on a two-dimensional detector such as imaging plates (IPs) by a single X-ray exposure, allowing for the simultaneous acquisition of normal and shear stresses [31].

Figure 13 shows the cos-α X-ray diffraction residual stress method apparatus. Before use, pre-program the tool with the operating conditions and set the specimen distance at a 45 mm angle. Next, the X-ray beam was used to calibrate the stress-free steel specimen. Since this diffractometer has no spinning moving parts, it is necessary to use a standard specimen to determine the location of its axis center. The reference specimen is a stress-free, resin-molded iron powder. With the proper settings, the residual stress in this specimen is in the region of 10 MPa, or almost nil, making the tool ready to use on additional specimens.

Fig. 13 The cos-α X-ray Diffraction residual stress method equipment [37], [38]

The relationship between normal stress components σij and the strain εϕΨ along the scattering vectors ϕ and Ψ in the case of elastically isotropic specimens is shown in Equation 1 and Figure 14, as follows:

$$\begin{aligned}\varepsilon_{\phi\psi} = {}^{1}/_{2}\, S_2\{(\sigma_{11}\cos^2\emptyset + \sigma_{12}\sin 2\emptyset \\ + \sigma_{22}\sin^2\emptyset)\sin^2\Psi + (\sigma_{13}\cos\emptyset \\ + \sigma_{23}\sin\emptyset)\sin 2\Psi + \sigma_{33}\cos^2\Psi\} \\ + S_1(\sigma_{11} + \sigma_{22} + \sigma_{33})\end{aligned} \tag{1}$$

where:

$\sigma_{11}, \sigma_{22}, \sigma_{33}$ = Normal stress in the specimen coordinate axes in X, Y, and Z directions.

σ_{23} = Shear stress in the Z axis directions normal to the Y axis.

σ_{12}	=	Shear stress in the Y axis directions normal to the X axis.
σ_{13}	=	Shear stress in the Z axis directions normal to the X axis.
S_1 and S_2	=	The X-ray elastic compliance constants. They can be related to young's modulus E and Poisson's ratio v of the body. $S_1 = -v/E$ and $S_2 = (1-v)/E$.

The strain $\varepsilon_{\phi\Psi}$ is defined in the following [40]:

$$\varepsilon_{\phi\psi} = \frac{d_{\emptyset\Psi}-d_0}{d_0} \tag{2}$$

where:

$d_{\emptyset\Psi}$	=	Lattice spacing along a scattering vector (the directions will be ϕ and Ψ).
d_0	=	Strain-free lattice spacing.

Because the strain-free lattice spacing varies greatly depending on the crystalline state, determining an exact value for d_0 is challenging. However, an optimization approach can calculate d_0 [37].

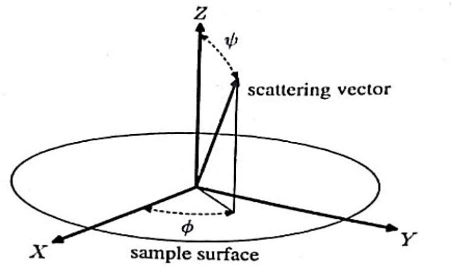
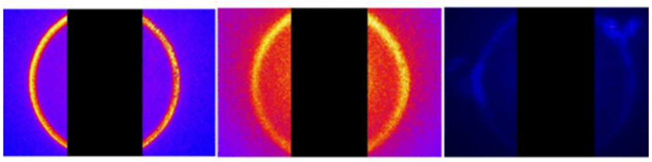


Fig. 14 A coordinate system for stress analysis through X-ray Diffraction [41]

The data obtained by the cos-α X-ray Diffraction method are classified into three classes based on a Debye-Scherer ring diffraction pattern, as shown in Figure 15. Only good and mediocre classifications were considered. The measurement findings reveal residual stress in directions 11 (σ11) and 12 (σ12), where σ11 is the perpendicular direction of the cross-section, and 12 is the parallel lengthwise direction of the cross-section. The crystalline lattice is the foundation of the X-ray Diffraction principle; a strong texture impedes data calculation, resulting in bad classification and a questionable result.



(a) (b) (c)

Fig. 15 X-ray intensity around the Debye-Scherer ring [37]

where:

(a) The distribution exhibits an even distribution, indicating a good classification.

(b) The data is scattered, yet some exhibit stronger than others. The data has a mediocre classification.

(c) Absent or very low intensity classified as bad data.

III. Results and Discussion

A. *Water Sanding Technique*

The uncoating technique with water sanding gives three classifications on a Debye-Scherer ring diffraction pattern: good, mediocre, and bad classification. Most of the results indicate a mediocre classification, while 9% to 20% indicate a bad classification. Figure 16 presents the results of measuring residual stress in cold-formed steel lip-channel sections using the cos-α X-ray Diffraction method with the water sanding technique.

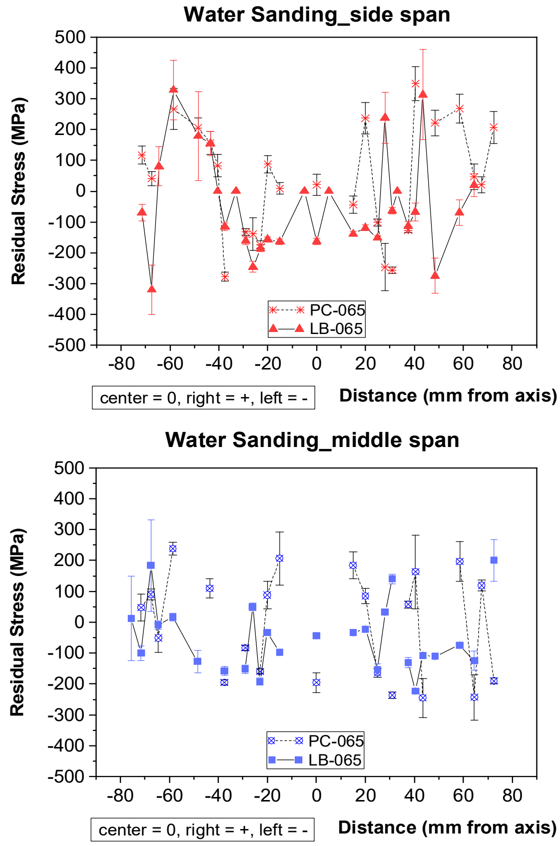


Fig. 16 The residual stress comparison between LB-065 and PC-065 using water sanding techniques. Side span (top); Middle span (bottom)

The PC-065 specimen's cold-formed steel-lipped channel sections exhibit a maximum residual stress of approximately 350 MPa on the side span and 329 MPa on the middle span. For the LB-065 specimen, the maximum residual stress is approximately 240 MPa on the side span and approximately 223 MPa on the middle span.

B. *Chemical Solutions Technique*

1) *The First Procedure:*

For the steel plate specimens, the average difference in dry mass between the bulk and surface for LB is 98,604%, and for PC, it is 98,662%, as shown in Figure 17 and Table 1. The observation also indicates that the chart remains sloping during the first 4 hours; however, after 4 hours, significant peeling begins. Figures 18 and 19 demonstrate that the LB02's coating outperforms the PC's, as indicated by the presence of some coatings on the LB after 17 hours when nearly all of the PC had peeled off.

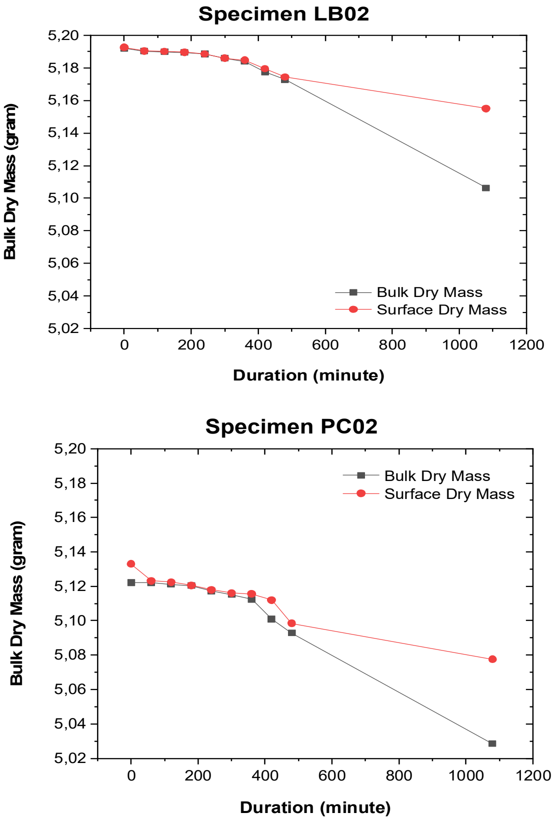


Fig. 17 Comparison of specimen LB (top) and PC (bottom)

TABLE I
COMPARISON BETWEEN LB AND PC

Date (May)	Starting Time	Duration (minute)	Bulk dry in a desiccator vacuum (gram)		Surface dry mass (gram)	
			LB02	PC02	LB02	PC02
	10.10	0	5,1919	5,1222	5,1925	5,1329
	11.10	60	5,1902	5,1221	5,1903	5,1232
23rd	12.22	120	5,1897	5,1212	5,1901	5,1224
	13.40	180	5,1894	5,1204	5,1895	5,1206
	15.13	240	5,1885	5,1174	5,1885	5,1179
	09.05	300	5,1859	5,1152	5,1859	5,1161
24th	10.32	360	5,1838	5,1123	5,1847	5,1156
	12.54	420	5,1775	5,1010	5,1794	5,1120
	14.35	480	5,1727	5,0929	5,1744	5,0985
25th	07.45	1080	5,1063	5,0285	5,1550	5,0775

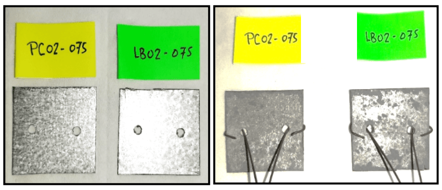


Fig. 18 The specimen LB and PC. Left - before coating; Right - after 8 hours of mixing

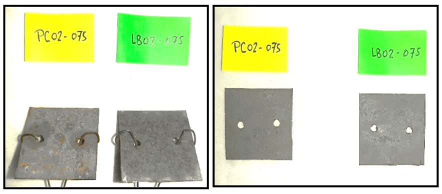


Fig. 19 The specimen LB and PC after 17 hours dipping (before and after ultrasound cleaning)

Figure 20 illustrates the liquid and specimen condition for the cold-formed steel lip channel C-section after 70 hours, as shown in picture 1. In picture 2, nearly all the dipping specimens have peeled off. In picture 3, remove the remaining dirt coating. Picture 4 shows the surface condition following a toothbrush and tap water cleaning. Pictures 4a, 4b, and 4c reveal some spots peeled off completely. After 70 hours of dipping, the specimen surface is still unsatisfactory enough to remove the coating. Next, repeat the dipping process for an additional 48 hours, as seen in Figure 21. The vacuum desiccator then receives the specimen to dry the surface. After that, the specimen is ready to measure.

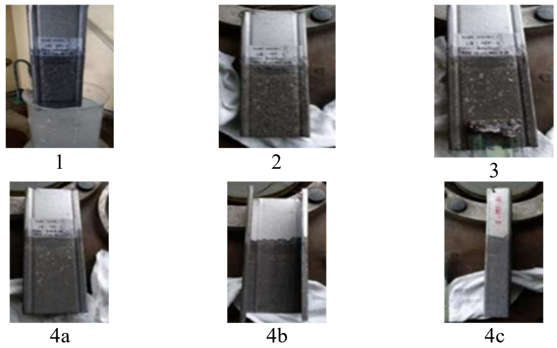


Fig. 20 The specimen results after a 70-hour dipping time

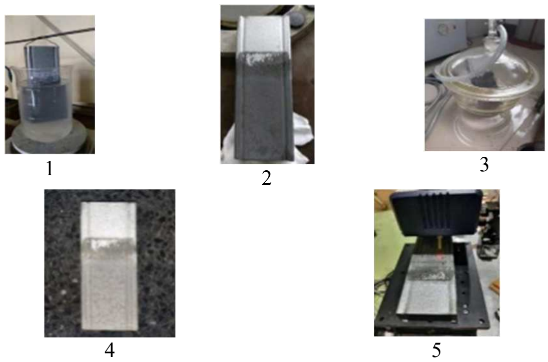


Fig. 21 The results of specimen observation after a 118-hour dipping time

2) The Second Procedure:

Explored methods to accelerate and enhance the uncoating results following the initial treatment, involving several experiments with various combination compositions that were significantly increased. This second procedure, which only used a 25% hydrochloric acid (HCl) solution, gave satisfying results. This procedure converts the coating into uncoated specimens, a process that takes approximately 4 minutes. Figures 22 and 23 show the uncoating results achieved using this procedure.

Fig. 22 The uncoating result for a 3 x 3 cm plate specimen after 4 minutes

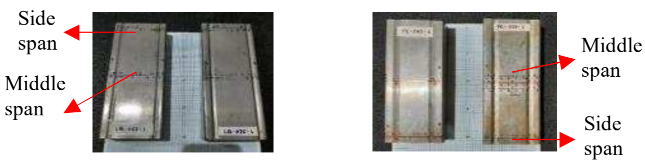
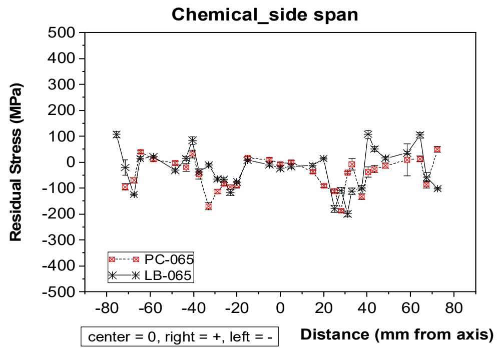


Fig. 23 The uncoating result for CFS lipped channel specimens

Figure 24 illustrates the residual stress comparison of the cold-formed steel lip channel C-section between the LB-065 and PC-065 specimens using a 25% hydrochloric acid (HCl) solution and chemical uncoating techniques. Based on the results, the PC-065 specimen's cold-formed steel-lipped channel sections have a maximum residual stress of 189 MPa on the side span and 187 MPa on the middle span. For the LB-065 specimen, the maximum residual stress is 199 MPa on the side span and 189 MPa on the middle span.



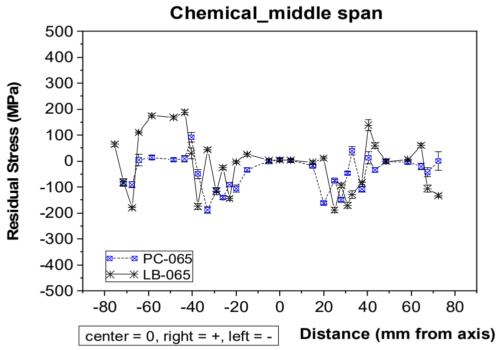


Fig. 24 The residual stress comparison between LB-065 and PC-065 using 25% hydrochloric acid (HCl) solution and chemical uncoating techniques. Side span (top); Middle span (bottom)

The following tables and figures compare the two uncoating techniques for measuring the residual stress in cold-formed steel using the cos-α X-ray Diffraction method.

TABLE II
COMPARISON COATING AND UNCOATING FOR SPECIMEN WITH 0,65 MM THICKNESS AND SIZE OF 3 X 3 CM

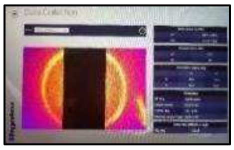
Observation	Coating	Uncoating
Mass		
Thickness		
Solution liquid condition		
XRD measurement intensity and results	 σ = 240,1 ± 120,2 MPa	 σ = -12,2 ± 6,4 MPa

TABLE III
COMPARISON COATING AND UNCOATING FOR LIP CHANNEL SPECIMEN OF LB

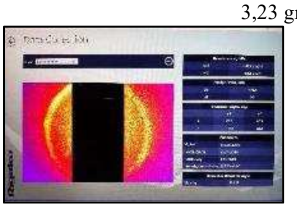
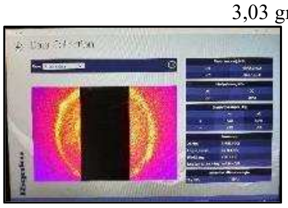
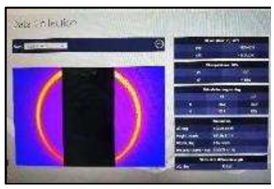
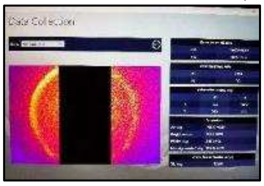
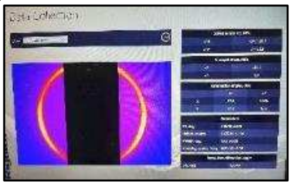
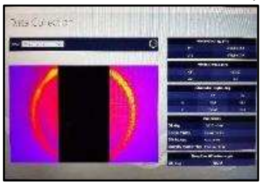
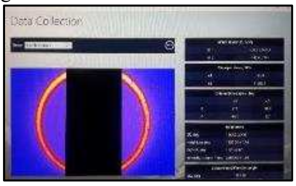
Specimen Code	**LB-065-1**		**LB-075-1**	
Observation	**Coating**	**Uncoating**	**Coating**	**Uncoating**
Mass (grams)	 145,78	 142,55	 165,75	 162,72
Mass deviation	3,23 grams		3,03 grams	
XRD measurement intensity and result	 σ = -148,6 ± 127	 σ = -38,6 ± 8,1	 σ = -197,9 ± 145	 σ = -56,0 ± 7,0

TABLE IV
COMPARISON COATING AND UNCOATING FOR LIP CHANNEL SPECIMEN OF PC

Specimen Code	**PC-065-1**		**PC-075-1**	
Observation	**Coating**	**Uncoating**	**Coating**	**Uncoating**
Mass (grams)	 144,23	 141,02	 168,48	 165,33
Mass deviation	3,21 grams		3,15 grams	
XRD measurement intensity and result	 σ = -144 ± 164,2	 σ = -24,1 ± 6,1	 σ = -172,4 ± 45,3	 σ = -33,1 ± 54,0

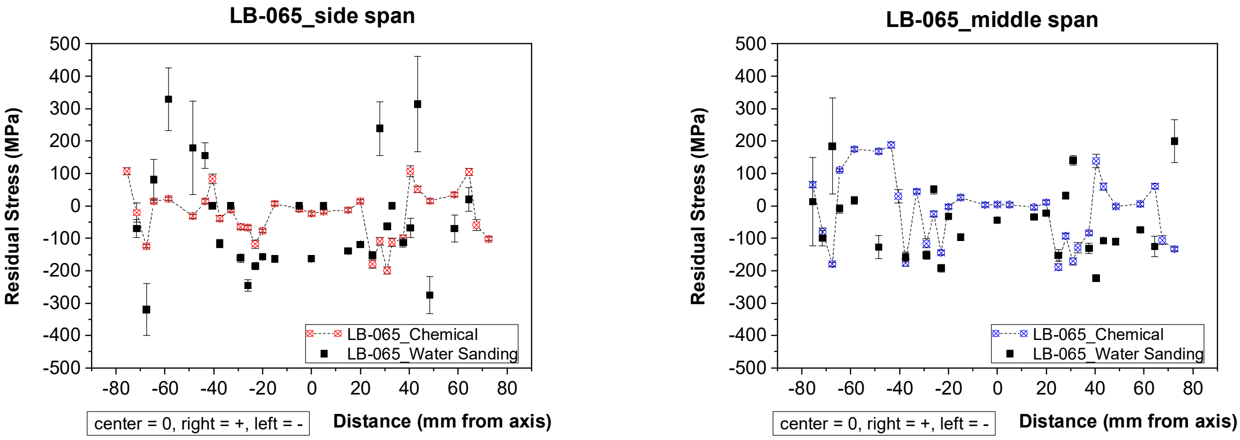


Fig. 25 The residual stress comparison between two uncoating techniques for PC-065. Side span (left); Middle span (right)

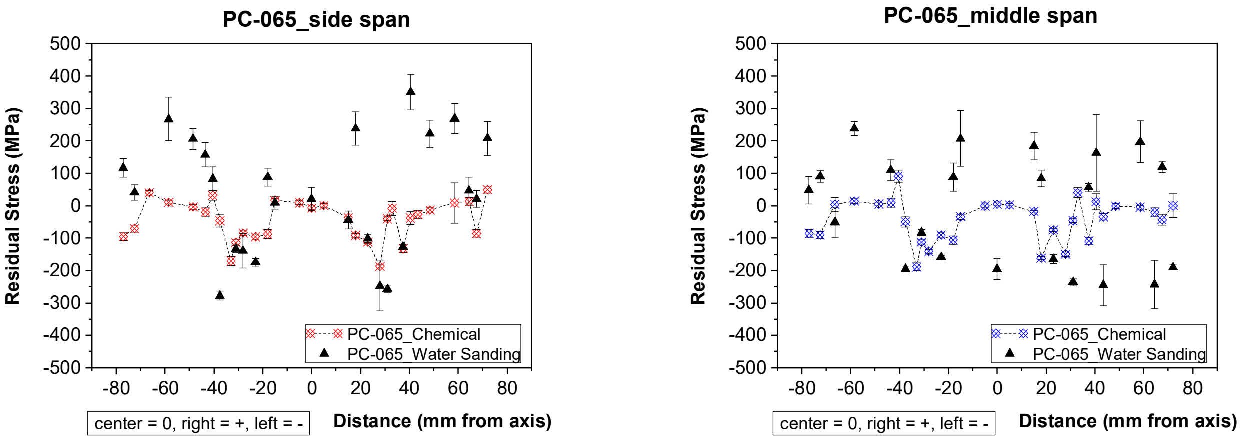


Fig. 26 The residual stress comparison between two uncoating techniques for PC-065. Side span (left); Middle span (right)

Table II delineates the distinctions between the coated and uncoated states of a 3 x 3 cm plate specimen. The coating thickness is 0.05 mm, as indicated on the specimen's mill certificate. Moreover, before the uncoating process, the specimen's mass was 4.4644 grams, whereas after the uncoating process, the mass diminished to 4.0829 grams. The mass deviation of the specimen is 0.3815 grams. The X-ray intensity surrounding the Debye-Scherer ring is a bad classification for coated specimens, while it is a good classification for uncoated specimens. The residual stress results for the uncoated specimen were reduced by over 100% compared to the coated specimen.

Tables III and IV indicate that the mass deviation for the lipped channel specimens, both LB and PC, is around 3 grams per specimen. The X-ray intensity surrounding the Debye-Scherer ring exhibited optimal distribution when measuring residual stress on the uncoated surface of cold-formed steel, revealing significantly lower and more rational values than the coated specimens.

Figures 25 and 26 compare the residual stress between two methods of uncoating: water sanding and a chemical solution containing 25% hydrochloric acid (HCl). The results show that the water sanding uncoating technique produces higher residual stress than the chemical solution uncoating technique, with approximately 50 to 160 MPa on the side span and approximately 10 to 50 MPa on the middle span.

IV. Conclusion

An experimental study of two uncoating techniques for measuring residual stress in cold-formed steel using the cos-α X-ray diffraction method has been presented. The first technique is water sanding, while the second involves a chemical solution. Two procedures are performed with the chemical solution: the first consists of combining a 25% hydrochloric acid (HCl) solution with a 25% ammonium hydroxide (NH_4OH) solution, while the second procedure utilizes a 25% hydrochloric acid (HCl) solution exclusively. The water sanding uncoating technique produces mostly mediocre results, while values ranging from 9% to 20% indicate a bad classification.

For the chemical uncoating technique, the first procedure that combines a 25% hydrochloric acid (HCl) with a 25% ammonium hydroxide (NH_4OH) solution shows a mediocre classification of the Debye-Scherer ring. One must control consistency and timing to achieve a flawless layer grinding result. On the other hand, using only a 25% hydrochloric acid (HCl) solution eliminates the coating on cold-formed steel. It provides a good classification of cos-X-ray diffraction intensity data related to the Debye-Scherer ring. The duration from coating to uncoating for each specimen dipped in the liquid is approximately 4 minutes.

Therefore, the results of the two methods show that the water-sanding uncoating method creates residual stress levels up to 160 MPa higher than those found with the chemical solution uncoating method. This is up to 29% greater than the yield stress of the cold-formed steel. Finally, removing the coating from the cold-formed steel surface is the key to successfully implementing the cos-α X-ray diffraction method.

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