Effect of Curing Temperature on Properties and Solvent Welding Strength of Ovalbumin

Yi-chuan Lin,[1] Z. C. Chang,[2] C. B. Lin,[1] Chien-Chin Shen,[1] Yen-Li Su[1]

[1]*Department of Mechanical and Electro-Mechanical Engineering, Tamkang University, Tamsui, Taipei, Republic of China*
[2]*Department of Mechanical and Electro-Mechanical Engineering, National Chin-Yi University of Technology, Taiping City, Taichung County, Republic of China*

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ABSTRACT: Effect of curing temperature on properties of ovalbumin has been investigated. Functional basis, crosslinking degree, tensile strength, and hardness increases with rising curing temperature. The increase of visible transmittance and the decrease of absorption accompany with increasing wavelength. The absorptive peak shows at 440–450 nm and the wavelength of the absorptive peak increases with the rising curing temperature. The relationship of joint strength with solvent welded joints of ovalbumin to their microstructure is also investigated. Ovalbumin can promote joint strength after the treatment of distilled water and curing. Comparing joint strength with fracture morphology, the smoother fracture surface morphology is related to the maximum tensile and shear joint strength, respectively. The joint strength is increasing with curing temperature and compressive stress, and the joint strength of treatment with 150°C curing temperature and 0.12 kgf/mm^2 compressive stresses are larger than its original tensile fracture strength of cured ovalbumin at same curing temperature. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 2144–2153, 2012

Key words: ovalbumin; crosslinking; curing; tensile; joint strength; solvent welding

INTRODUCTION

Curing treatment is one of the most promising approaches to improve the gelling properties of ovalbumin. According to Vollrath,[1] compare with nonheated OVA (mainly consisted of egg-white protein), the dry-heated OVA are forming clearer and firmer gels. In other words, the properties of ovalbumin can be improvement by heating in dry state.[2] According to Divair et al.,[3] the gel mechanical properties and network density of the ovalbumin showed a strong dependence on heat treatment temperature (70, 80, and 90°C). The equilibrium stress of egg-white and ovalbumin gels using the statistical theory of rubber elasticity suggested that 3.9 covalent disulfide crosslinks per ovalbumin molecule were formed on gelling.[4] Juana et al.[5] showed that although, 55°C was the temperature for starting to coagulate in both types of egg-white, only in hen egg-white was the process completed at 65°C. The gels obtained at 80°C (both ostrich and hen egg-white gels) presented a strong structure that would allow ostrich egg-white to be used as a gelling agent in the food. Most of researches probed the gel and rheology properties of the ovalbumin went through curing treatment, the present study of the first part further studying the effect of curing treatment on optical hardness and tensile property of ovalbumin.

The application of thermoplastic polymers' solvent welding (polystyrene, poly(methyl methacrylate), and poly(vinyl chloride) is well known. For example, poly(methyl methacrylate) treated with solvent welding by chloroform in the signboard. To promote welding strength, the solvent is required to plasticize[6] and decrease the glass transition temperature[7] of welding surface strongly, i.e., to increase the rate and the extent of interdiffusion of the polymer chains, not to transfer load from one adhered to the other, for example tetra hydrofuran (THF) and cyclohexanone (CYH) is a good solvent for poly vinyl chloride (PVC) to produce excellent welding strength, but methyl ethylkefone (MEK) is a poor one producing a poor welding strength[8]; polycarbonate treated with butanone has maximum welding strength than treated with acetone or cyclohexanone.[9] Cherry and Evely,[10] showed that a solvent affected zone (SAZ) is formed by the welded thermoplastic polymer after solvent welding and the residual solvent concentrated in the solvent affected zone.[11] According to Menges et al.,[12] a soft residual final adhesive layer (FAL) sandwiched between two

Correspondence to: C. B. Lin (cblin@mail.tku.edu.tw).
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TABLE I
Various Composition of Ovalbumin

Protein	Content (%)	Isoelectric point (pI)
Ovalbumin	55.9	4.8–4.9
Conalbumin	14.2	5.6–6.1
Ovomucoid	12.7	4.2–4.8
Lysozyme	3.8	10.2–11.6
G_2 globulin	4.3	6.6
G_3 globulin	4.5	5.3
Ovomucin	4.5	–
Avidin	0.1	~10

solvent-affected zones (SAZ). Yue,[8] showed that the solvent welding strength is depend on the compatibility between solvent and polymeric materials, but the size of the SAZ is independent, based on the model of critical principal strain.[13]According to Wool and O'Connor,[14]welding of polymer interfaces involving surface rearrangement, surface approach, wetting inter, diffusion, and randomization, which the wetting and interdiffusion are the main parameters of controlling welding strength. Fractography relates to the topography of the fracture surface to cause or base the mechanistic fracture.[15] The fractography of solvent welding of poly(methyl methacrylate) and polycarbonate was investigated by Lin et al.[16] and Chang et al.[9] Little study was done on the effects of curing temperature on solvent welding of the ovalbumin, the purpose of the second part research was to determine whether the solvent welding strength of the ovalbumin could be increased by curing treatment. The relationship of solvent welding strength to fractography was also investigated. Finally, a conclusion will be made.

MATERIALS AND METHODS

Materials preparation

The ovalbumin was extracted from eggs of Legthron and salting out purified its solution of it. The ingredients of the ovalbumin are listed in Table I. In sample fabrication, the glass ball was placed in ovalbumin mixture liquor at first and then vibrated by ultrasonic wave for 90 min to form homogenized ovalbumin mixture liquor. The process of homogenizing can solve the problem of a ripple defect brought to the sample through curing. Then the homogenized ovalbumin liquor was desorbed at the relative humidity of 80% until there was no change in weight under the measurement of a digital balance (±0.1 mg). The sample of ovalbumin was obtained eventually. Desorption processing was made under the relative humidity (80%) to prevent the sample from bending during the process. Pieces of dimensions 30 × 20 × 2 mm^3 cut from the ovalbumin planking sheet were used for spectrum analysis, and the gauge length 10 mm for tensile test. The samples were cured in air for 30 min at 90, 120, and 150°C (±2°C), and then the samples were polished on a 2000 grid emery paper, and polished with lubricant and 0.05 μm alumina.

FTIR spectroscopy

Curing samples' powder were mixed with potassium bromide and pressed into the form of pellets. The cured specimen of Fourier transform infrared (FTIR) spectra was measured in the range 400–4000^{-1}cm by the attenuated total reflectance method using a Bio-Rad FTS-135 FTIR spectrometer.

Spectrum measurement

Transmittance of cured specimen was measured in the wavelength range of 390–780 nm using a Hitachi U-3410/U-3240 spectrometer. The scanning rate was 1200 nm/min.

Mechanical properties

The cured specimen was mounted on a tensile test machine and tested at a crosshead speed of 1 and 2 mm/min at room temperature. To study the effect of cold work on tensile properties, the tensile sample with curing temperature of 90 and 120°C was tensile under room temperature and at a 2 mm/min crosshead tensile speed until a given constant cold work then release it, then tensile it again until it was broken. The morphologies of the fracture surfaces of specimens tested in the various tests were observed with a LEO-1530 scanning electron microscope (SEM), following gold plating of 2 min before observation.

A MATSUZAWA SEIKI Co. MvV-6151 Vickers hardness machine was used to test the hardness

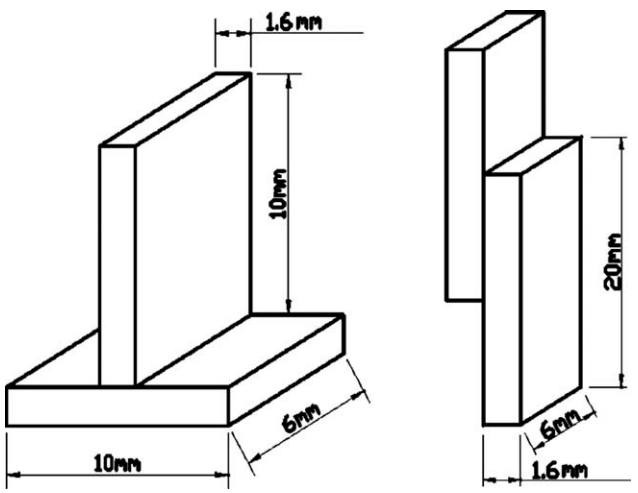

Figure 1 Schematic diagrams of various joints to test solvent welding: (a) tensile test; (b) shear tests.

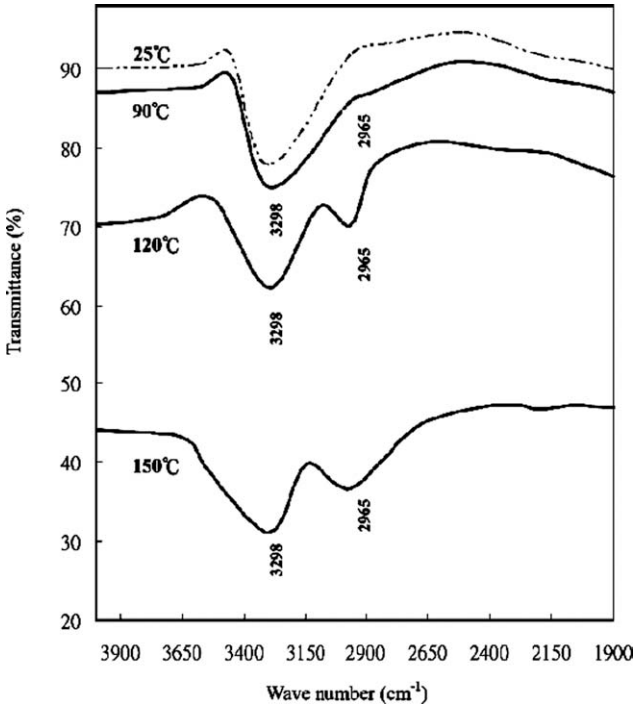

Figure 2 The FTIR spectra of ovalbumin at 25, 90, 120, and 150°C curing temperature.

under the loading of 1 kgf, with the loading time of 30 s and the loading rate of 45 μm/s. This was measured for 5 times at different positions and the average hardness of various cured temperature samples was obtained.

Solvent welding

Distilled water was employed as solvent for solvent welding. The distilled water 2 ml was wetted to the surface of the ovalbumin at 25°C. The two welding surface were brought together and the specimen was mounted under a compressive stress of 0.06 kgf/mm^2 and 0.12 kgf/mm^2 for 1 min. Then the welded specimen was cured in air at 90, 120, and 150°C for 30 min. Pieces of dimensions 10 × 6 × 1.6 mm^3 for tensile tests and 20 × 6 × 1.6 mm^3 for shear tests. The specimen geometries for tensile and shear tests are shown in Figure 1(a,b). According to Titow et al.[11] tests of solvent welding of plastics, the smaller the concentration of solvent after desorption,

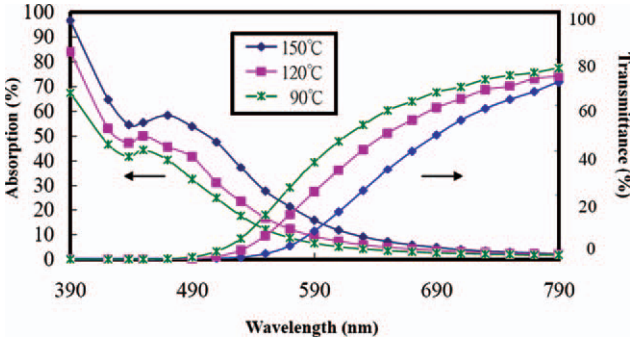

Figure 3 The absorption and transmittance spectrums of ovalbumin at 90, 120, and 150°C curing temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

the smaller is the diffusivity. Hence, residual solvent remains after welding, and decreases the joint strength. It is worthy mention that the welded of the ovalbumin after curing treatment, without residual solvent remains in the welded specimens.

Specimens for tensile and shear tests were mounted on a universal tensile testing machine with a crosshead speed of 5 mm/min, tests were run at 25°C. Shear and tensile strengths are defined as failure loads divided by welded area. Each data point was the average of three specimens. The morphologies of the fracture surfaces of specimens tested in the various tests were observed with a LEO-1530 SEM, following gold plating of 2 min before observation.

RESULTS AND DISCUSSION

Functional basis and color

The FTIR spectrum of the ovalbumin treated with and without curing is illustrated in Figure 2. The spectrum of ovalbumin without curing treatment shows the absorption peak of amine group at 3298 cm^{-1}, and the vibration bands of alkyne's group at 1640–1680 cm^{-1} and 1350—1470 cm^{-1}, respectively. Comparing the cured ovalbumin with the ovalbumin without curing treatment, some absorption peaks have shifted a little and their intensities were reduced, but there was no new intense peak could be observed. Additionally, because of a higher curing temperature provided more energy to decompose

TABLE II
Tensile Properties of Various Curing Temperatures

Curing temperature (°C)	$\sigma_{y0.2\%}$ (Kg$_f$/mm^2)	UTS (Kg$_f$/mm^2)	σ_f (Kg$_f$/mm^2)	E (MPa)	ε_f (%)
90	21.8	26.1	24.0	3.7	–
90[a]	6.6	8.7	4.3	12.4	1.4
120	20.6	30.3	27.4	6.4	–
120[a]	34.1	40.8	34.0	7.7	0.9

[a] Second cyclic tensile testing.

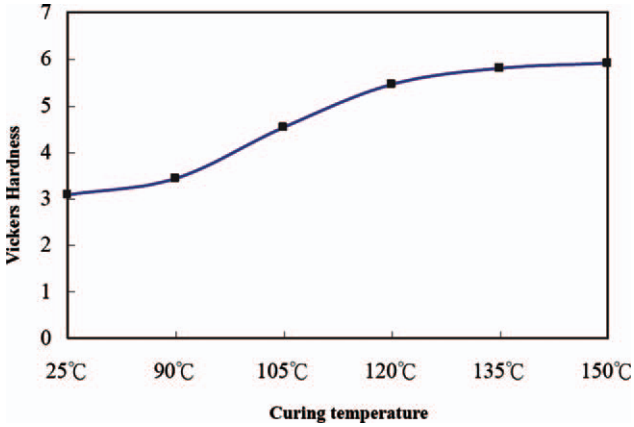

Figure 4 Vickers hardness versus curing temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

molecules and diffuses higher consistency of oxygen, the curing temperature increased the transmittance is reduced. Under the action of thermal-oxygen decomposition, the ovalbumin molecules decomposed more free radicals. During the process of thermal decomposition, free radicals and adjacent molecules led to scission effect forming more free radicals. More unsaturated free radicals absorbed more infrared rays, causing transmittance to decrease.

The optical spectrums of the cured ovalbumin are shown in Figure 3. Within the range of visible light (390–780 nm), transmittance rose as wavelength increased, went down as curing temperature decreased and the transmittance approached zero as the wavelength was between 390 and 450 nm. Comparatively, the absorption decreased and approached zero as the wavelength increased. As for 440–450 nm, there was an absorption peak whose absorption rose as curing temperature increased. The reason

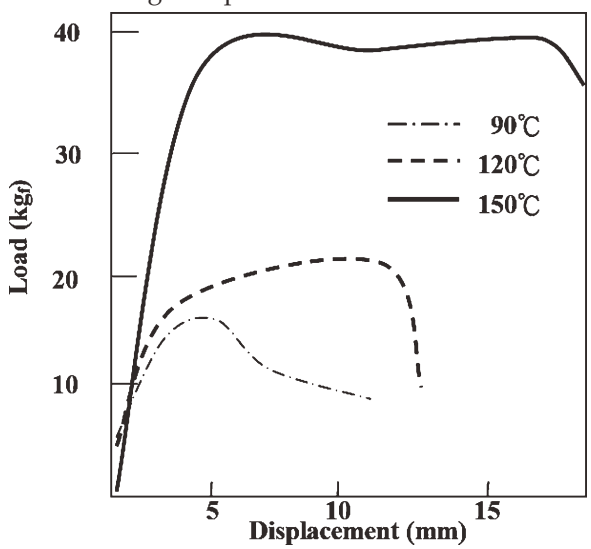

Figure 5 Influence of curing temperature on load-displacement curves for ovalbumin deformed in tension at crosshead speed of 2 mm/min.

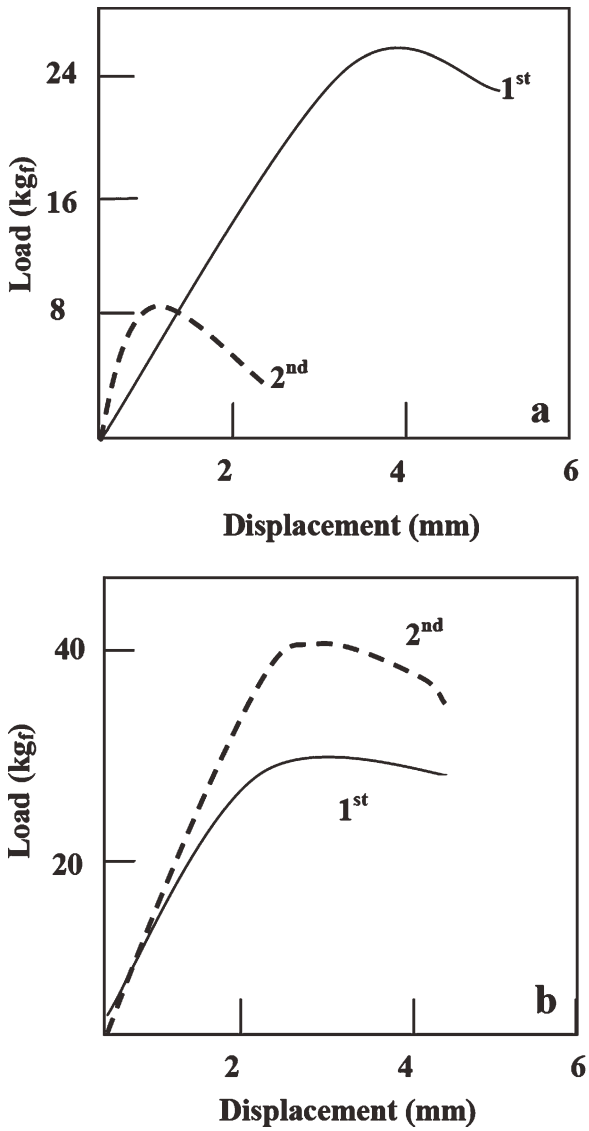

Figure 6 Influence of curing temperature on work hardening curves for ovalbumin deformed in tension at crosshead speed of 2 mm/min at 90 and 120°C curing temperature.

was that after visible lights entered the curing ovalbumin, except reflex light, partial energy would be absorbed by the unsaturated free radical in the cured ovalbumin and went up into an excited state and emitted specific absorption spectrum and visible light (brown) as went back to ground state. Because the number of unsaturated free radicals increased as curing temperature rose, relatively more photons were absorbed. It made transmittance lower or colors darker. When its curing temperature was 150°C the color was dark brown. What is worth mentioning that is if ovalbumin was placed in an oxygen-free system at 150°C curing temperature for 30 mins, the effect of thermal-oxygen decomposition reduced, the colors of the ovalbumin and the ovalbumin without curing stayed that was the same? This is because there was no free radicals activation-oxygen to

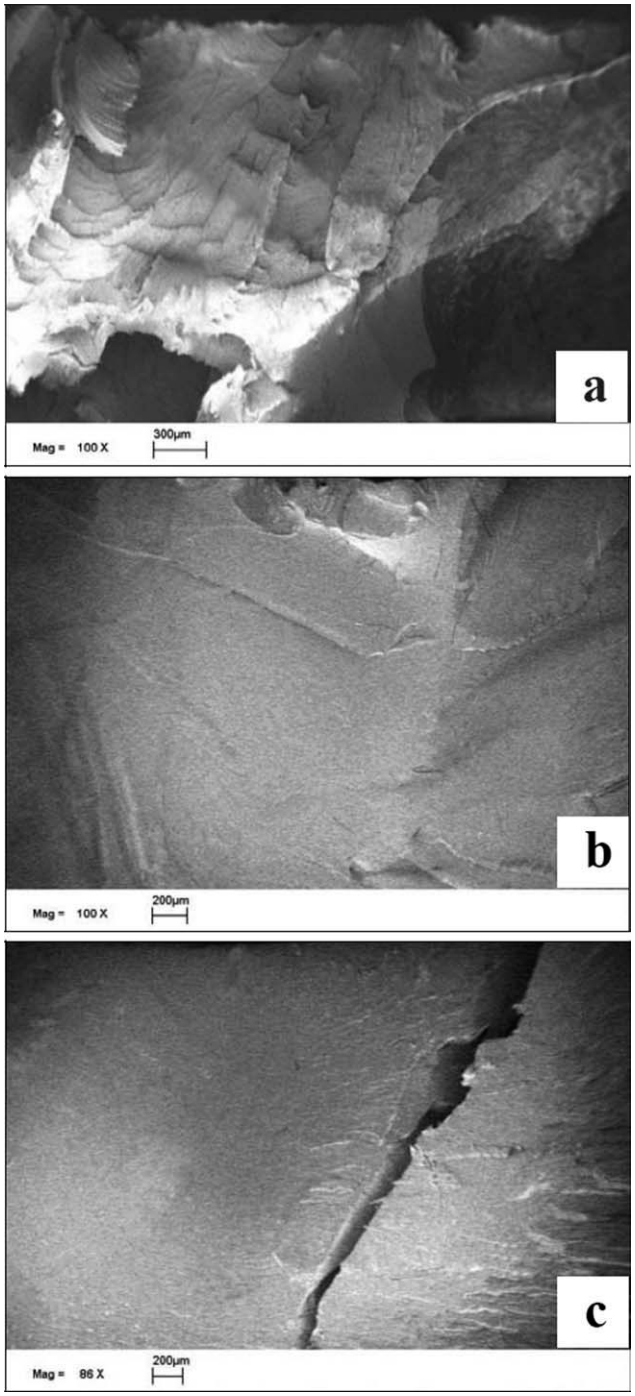

Figure 7 Micrographs of tensile fracture surface at (a) 90°C, (b) 120°C, and (c) 150°C curing temperature.

scission ovalbumin molecules; therefore production of free radicals was reduced.

Hardness and tensile properties

At 90–150°C curing temperature, the hardness of curing ovalbumin for 30 min rose to saturation state as curing temperature increased, as shown in Figure 4. The reason is that during the heating process, the unsaturated free radicals of ovalbumin were close to each other, so the activated oxygen free radicals and unsaturated free radicals resulted in crosslinking. However, for the pairs of free radicals at a further distance from each other and independent free radicals, it is more difficult to form crosslinkage. Therefore, the sample with lower curing temperature would remains with some unsaturated free radicals, which did not form crosslinking. Under certain oxygen pressure, free radicals increased as the curing temperature increased, and so did the interaction with activated oxygen's free radical. Therefore, crosslinking degree approached saturation as curing temperature increased. As a result of higher crosslinking degree, compressive and deformable resistance is greater better and so the value of hardness increased and approached saturation as curing temperature rose.

With different curing temperatures, the characteristic curve of tensile load and displacement are shown in Figure 5. At 120 and 150°C curing temperature, typical, hard, and tough macromolecules all

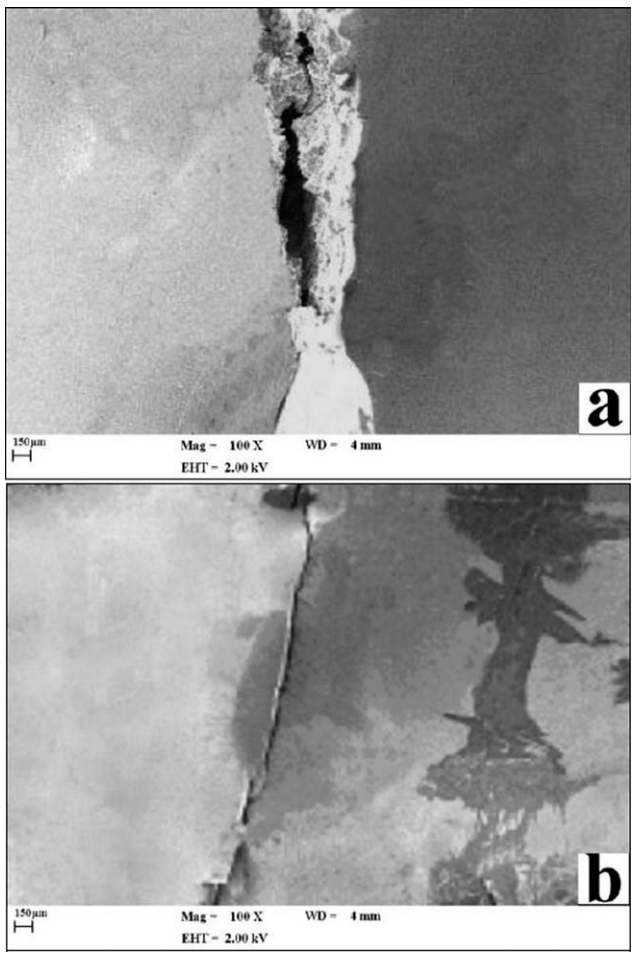

Figure 8 The morphologies of solvent affected zone of the ovalbumin treated with 90°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2;(b) 0.12 kgf/mm^2.

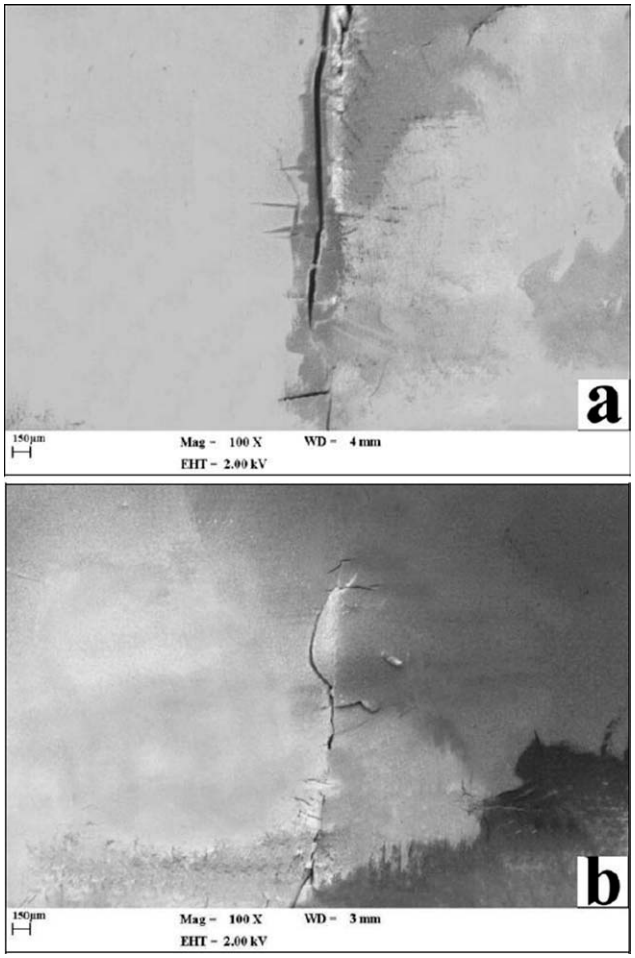

Figure 9 The morphologies of solvent affected zone of the ovalbumin treated with 120°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2; (b) 0.12 kgf/mm^2.

contain deformed action of uniformly necking. At 90°C curing temperature, an obvious yield point occurred and local necking deformation happened after maximum loading. The tensile strength rose as curing temperature increased, because the tensile strength is directly proportion to the crosslinking degree after curing. And the crosslinking degree is at its maximum at 150°C curing temperature. In Figure 5, after the load of 90°C cured samples reached its maximum, loading decreased in a slow gradual mode until it broke. Since, there was local necking occurred on the sample after maximum loading. Load decreased gradually not rapidly after local necking happened. Relative to the characteristic curve of 120 and 150°C curing temperature load-displacement, like the tensile results of several hard leather, there is a transition existing between linear section to nonlinear one. It shows obvious ductility in the process and uniform necking when the maximum load is exceeded. It is worth mentioning that the elongation of uniform necking at 150°C is greater than at 120°C.

Specimen at 90 and 120°C curing temperature were offered the same loading before they broke, then given stress release, extended stretched again to break. Its properties of tensile and characteristic curve of load-displacement are shown in Table II and Figure 6. In Table II, elastic modulus of second tensile is bigger than the ones of first tensile at the same curing temperature, according to the physics meaning of elastic modulus if it is able to resist fully plastic deformation by external force. Therefore, when first tensile is not broken, external force made the molecular chain of soft region orientation along the tensile direction and internal residue stress exited in hard region; when the second tensile process, it needed more external force to cause deformation of the molecular "roving-like" of the internal-stressed hard region and orientated soft region. $\sigma_{y0.2\%}$, UTS and σ_f of second tensile were smaller than that of first tensile at 90°C curing temperature; contrarily, $\sigma_{y0.2\%}$, UTS and σ_f of second tensile were greater than that of first tensile at 120°C curing

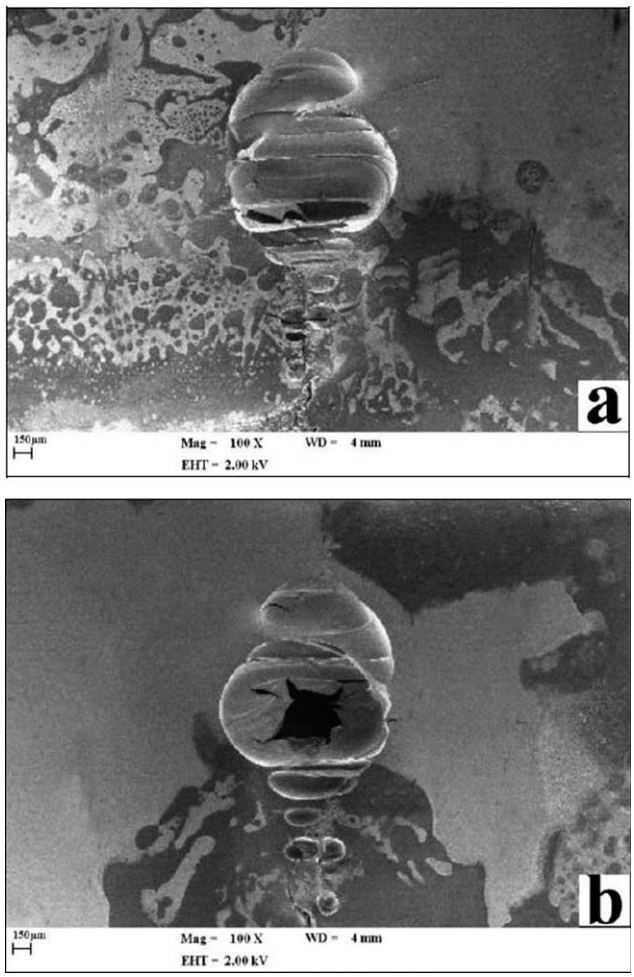

Figure 10 The morphologies of solvent affected zone of the ovalbumin treated with 150°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2; (b) 0.12 kgf/mm^2.

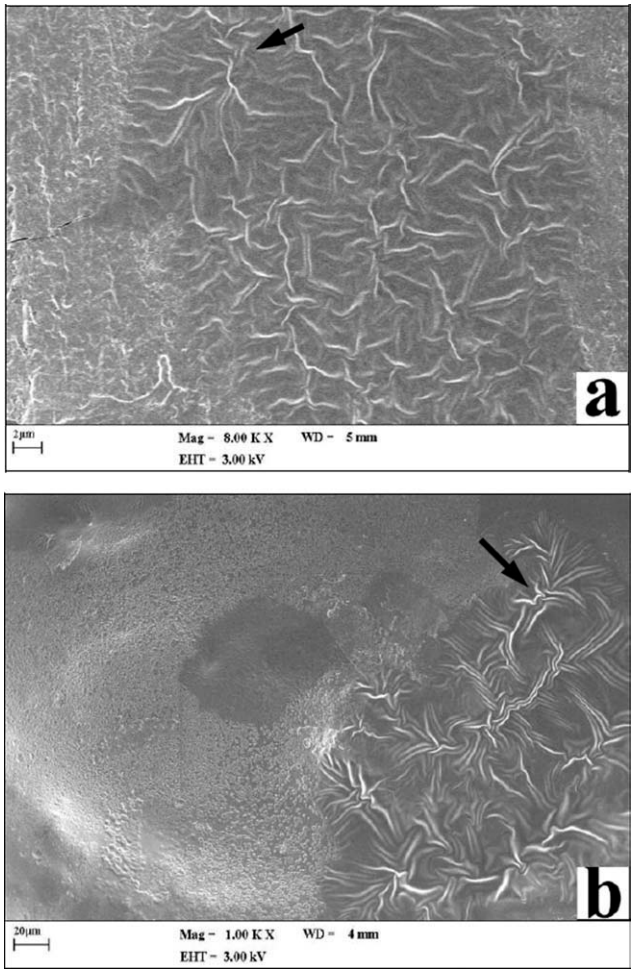

Figure 11 The morphologies of tensile fracture surfaces of butt joints of the ovalbumin treated with 90°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2; (b) 0.12 kgf/mm^2.

temperature. Although, the orientation happened at soft region in first tensile at 90°C, but partial entangled molecular chains of soft region were loosed and there were less hard region at 90°C. Specimen could not ensure the greater part tensile stress. Hence, when second tensile, the tensile stress only resisted partial entanglement of molecular chain and crosslinking of hard region to break. On the other hand, since soft region was less than hard region at 120°C curing temperature, when the first tensile molecular chains of hard region resisted deformation of sample, those entangled molecular chains of soft region were loosed. At the same time, partial entangled molecular chains had the chance to orientate. It needed more external force to overcome the molecular chains of hard region and orientated soft region when the second tensile.

The fracture morphology of different curing temperature is shown in Figure 7. The morphology of tensile fracture belonging to hybrid-type is somewhat tough and brittle, fracture morphology was smooth at higher curing temperature, but it was more obvious that the tough morphology at lower curing temperature. The fracture morphology of 120°C specimens was between 90 and 150°C.

Solvent affected zone

When both welding surface of a polymeric material are treated with a good solvent in the absence of an extraneous adhesive, the two welding surfaces are brought together under a suitable pressure, the SAZ in formed in the welded points and a soft residual FAL was sandwiched between two SAZ. It is because of the solvent affected zone arises from the "swelling" of polymeric polymers, thus the boundary between the adhered and solvent affected zone is clearly marked by the depth of solvent penetration into the polymer.

The solvent affected zone of the ovalbumin treated at 90, 120, and 150°C curing temperature are shown in Figures 8–10. They have found that, no obviously SAZ is formed in the welded parts, but the FAL is formed because of the hydrophilic ovalbumin and

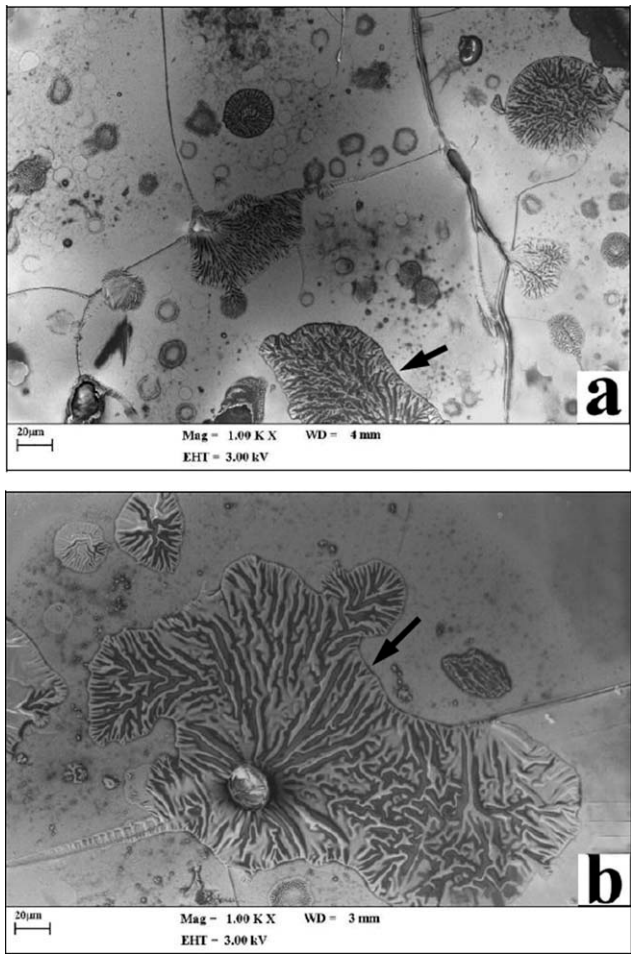

Figure 12 The morphologies of tensile fracture surfaces of butt joints of the ovalbumin treated with 120°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2; (b) 0.12 kgf/mm^2.

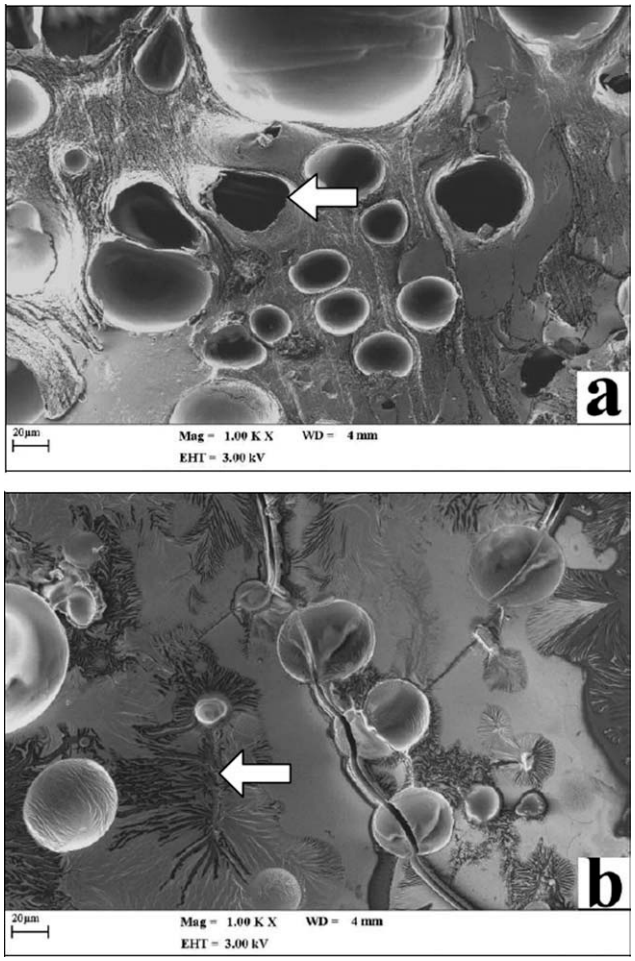

Figure 13 The morphology of tensile fracture surfaces of butt joints of the ovalbumin treated with 150°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2; (b) 0.12 kgf/mm^2.

the ovalbumin can be directly dissolved in the distilled water, and it generates a tacky-layer on the welding surface. The tacky-layer is squeezed easily while compressive stress is exerted on the welded parts, thus decrease the extent of solvent welding zone. The welding area increases with increasing curing temperature and compressive stress, due to the tacky substance in the FAL which is squeezed easily while compressive stress is exerted on it, and also help the molecular chains of welding surface approach, wet and interdiffusion each other. Moreover, the curing temperature can be helping the molecular chains in the FAL zone which produced crosslink. Note that the region containing the irregular holes in the tacky layer at 150°C curing temperature is shown in Figure 10, because of the crosslink of molecular chains in the FAL has been formed during curing treatment, but the 150°C curing temperature is above the boiling points of distilled water, thus the residual distilled water can be volatized from the FAL during curing treatment. When the stream volatized from crosslinking zone in the FAL will be induced larger internal pressure and some irregular holes are formed along the central line. As for 90 and 120°C curing temperature, the welded area and crosslinking degree is smaller than 150°C one, thus the volatized stream easily escape from the unwelded region, and the irregular holes cannot easily form in the FAL.

Fracture morphology

Figure 11–13 illustrate the morphologies of tensile fracture surfaces of butt joints of the ovalbumin treated with 90, 120, and 150°C curing temperature under 0.06 and 0.12 kgf/mm^2 compressive stress. The common feature of the tensile fracture surface has some "roving-like" morphology is formed in the partial zone, as indicated by arrow. The reason of roving-like morphology is formed that the distilled water can easily interact with the hydrophilic ovalbumin within the ovalbumin, and generates a tacky-layer on the welding surface, where the tacky region

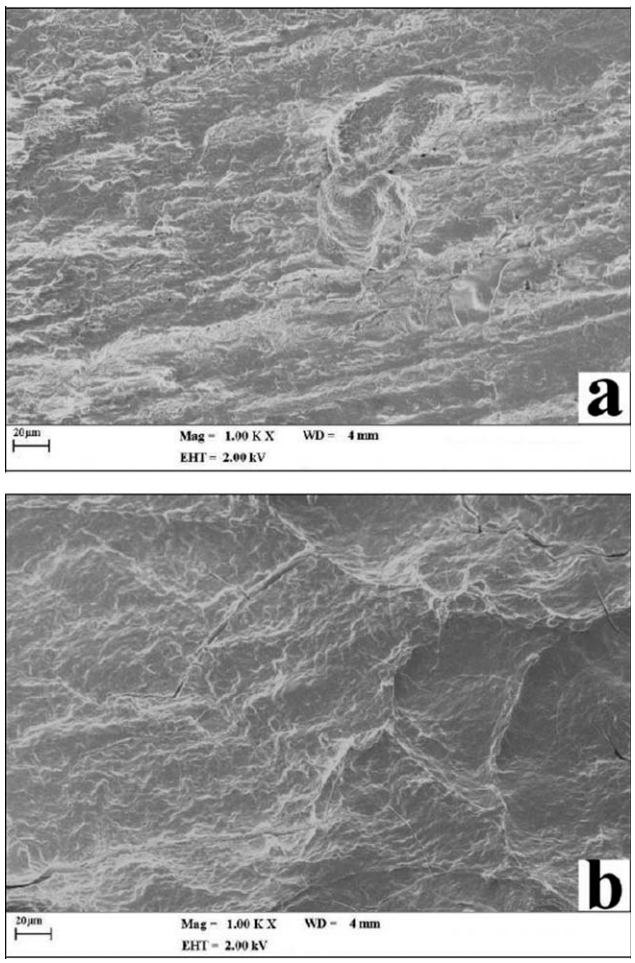

Figure 14 The morphologies of shear fracture surfaces of lap joints of the ovalbumin treated with 90°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2; (b) 0.12 kgf/mm^2.

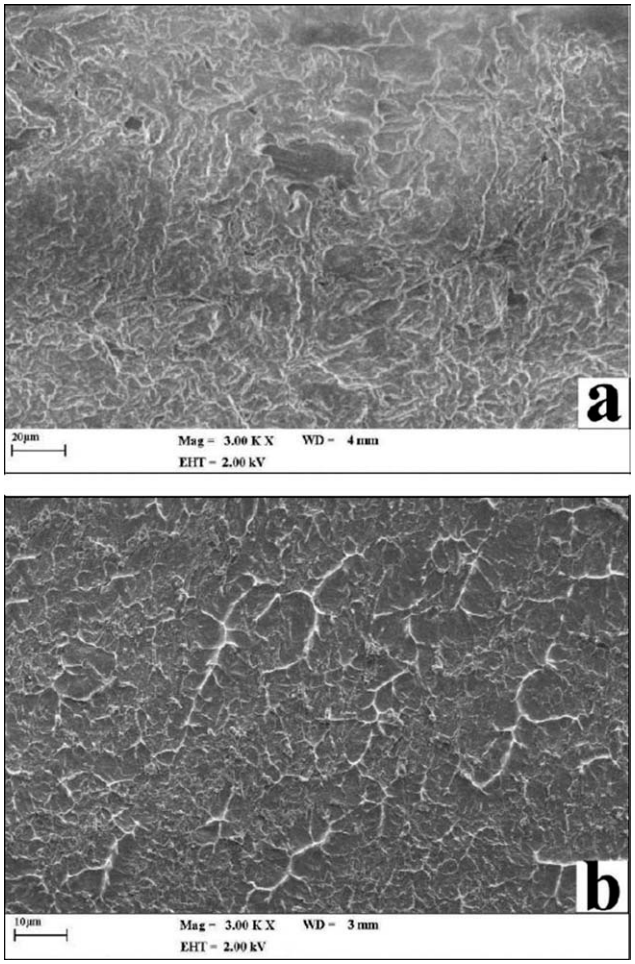

Figure 15 The morphologies of shear fracture surfaces of lap joints of the ovalbumin treated with 120°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2; (b) 0.12 kgf/mm^2.

should be produce crosslinking after curing treatment. When the welded part is tensile, the crosslinking region can sustain the larger tensile stress. In the mean time, the chains of hydrophilic ovalbumin should be oriented along tensile direction, and than the roving-like is formed. The curing temperature can increase the extent of crosslink of molecular chains, the entanglement degree of the roving-like increase with increasing curing temperature.

Comparing the morphology of the butt fracture surface with the solvent affected zone, the area of the roving-like morphology increases with compressive stress; the larger wetting area can increase interaction of the molecular chain of hydrophilic ovalbumin. In the meantime, we can also see that some holes are formed in the tensile fracture surface at 150°C curing temperature is arrow shown. Additional, it is found that the entanglement and the area of the roving-like increases with increasing applied compressive stress, and it exist obviously "knot" of the roving-like, because the compressive stress increase the extent of interdiffusion of molecular chains, and the holes arise from volatile of distilled stream. The morphologies of shear fracture surfaces of lap joints of the ovalbumin treated with 90 and 120°C curing temperature under 0.06 and 0.12 kgf/mm^2 compressive stress as shown in Figures 14 and 15. The common feature of shear fracture surface is "mountain-ridge like" morphology, and the extent of the mountain-ridge like increases with increasing curing temperature and compressive stress. The reason of mountain-ridge like morphology is that the mountain-ridge region is just interaction zone of the hydrophilic ovalbumin, when the welded part is sheared, the region produces shear deformation. Finally, the welded joint break in the mountain top as shown in white line region. The curing temperature and compressive stress can promote interaction of molecular chain in hydrophilic ovalbumin, thus the extent of the mountain-ridge like increase with curing temperature and compressive stress. The morphology of shear fracture surfaces of the ovalbumin treated with 150°C curing temperature under 0.06 and

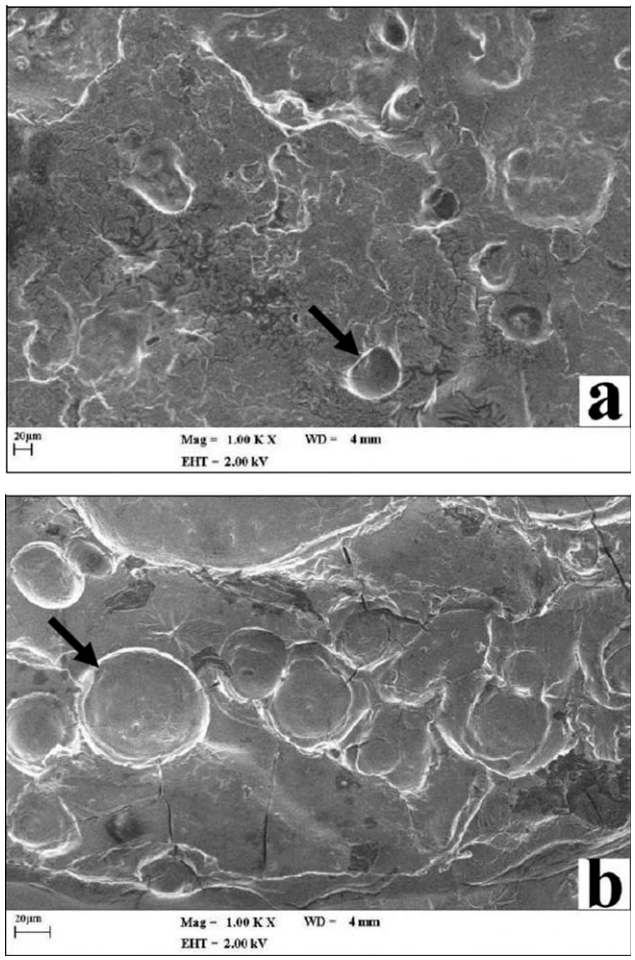

Figure 16 The morphologies of shear fracture surfaces of lap joints of the ovalbumin treated with 150°C curing temperature under compressive stress of (a) 0.06 kgf/mm^2; (b) 0.12 kgf/mm^2.

TABLE III
Welding Strength and Recovery of Various Curing Temperature

Joint model	Compressive stress (kg_f/mm^2)	90°C, σ_f (kg_f/mm^2)	120°C, σ_f (kg_f/mm^2)	150°C, σ_f (kg_f/mm^2)
Butt	0.06	0.12 ± 0.04	0.26 ± 0.01	0.32 ± 0.04
	0.12	0.17 ± 0.03	0.48 ± 0.05	0.73 ± 0.03
Lap	0.6	0.11 ± 0.02	0.28 ± 0.02	0.41 ± 0.01
	0.12	0.15 ± 0.02	0.34 ± 0.01	0.63 ± 0.06

0.12 kgf/mm^2 compressive stress as shown in Figure 16. The morphology of shear fracture surfaces has not found the mountain-ridge like, and replace the morphology is smoother fracture surface with some holes, because of this fracture surface is type of brittle fracture comes from the ovalbumin treated at 150°C curing temperature with heavy crosslinking.

Joint strength

The tensile and shear strength of butt and lap joints of the ovalbumin treated with 90, 120, and 150°C under 0.06 and 0.12 kgf/mm^2 compressive stresses are listed in Table III. It can be seen that whatever butt or lap fracture stresses all increases with increasing curing temperature and compressive stress. However, the tensile strength of these joints are about 71–149% that of the bulk tensile strength of the cured ovalbumin at corresponding to curing temperature. It is because of the compressive stress and curing temperature can be increased interdiffusion and crosslinking of molecular chains, respectively. Comparing the morphology of the butt fracture surface and SAZ with the corresponding tensile fracture stresses, we find that the larger density entanglement of roving-like and the smoother tensile fracture surface corresponding to the higher tensile strength. In comparing the fracture surface morphology and the shear strength, it is seen that the smoother shear fracture surface corresponds to higher shear strength.

CONCLUSIONS

The conclusion could be presented as following:

1. The numbers of functional basis, crosslinking degree, and hardness rose and approached saturated as curing temperature increased.
2. Transmittance rose as curing temperature decreased, and the transmittance approached zero as it was between 390 and 450 nm. There was an absorptive peak at 440–450 nm and the absorption increased while the curing temperature rose.
3. Yield stress, tensile strength, Young's modulus, and degree of work hardening all increased as curing temperature rose.
4. The effect of cold work-hardening at 120°C curing temperature was more obvious than at 90°C curing temperature.
5. Tensile-fracture morphology possessed the form between tough and brittle, brittle-fracture morphology increased as the curing temperature rose.
6. The welding area of the solvent affected zone and joint strength increasing with curing temperature and compressive stress.
7. Ovalbumin solvent-welded at 150°C curing temperature and 0.12 kgf/mm^2 compressive stress recovers a larger fraction of its original of cured ovalbumin.

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