Journal of the KSTLE Vol. 25, No. 1, February 2009, pp. 31~37
The Korean Society of Tribologists & Lubrication Engineers

The Roles of Ingredients in Fade Resistance of Brake Friction Materials

Keun Hyung Cho, Jae Min Han*, Sung Bin Park, Min Wook Shin and Ho Jang†
Dept. of Materials Science and Engineering, Korea University
**Materials Research Team, Advanced Technology Center, R&D Division for Hyundai Motor Company and Kia Motors Corporation*

자동차용 마찰재 원료가 열화 저항성에 미치는 영향

조근형 · 한재민* · 박성빈 · 신민욱 · 장 호†
고려대학교 신소재공학부, *현대자동차 연구개발본부 금속재료연구팀

(Received November 15, 2008 ; Revised December 22, 2009; Accepted December 31, 2008)

Abstract – 상용화 마찰재를 사용하여 고온 열화에 대한 원료들의 영향을 연구하였다. 제한된 영역에서의 실험계획법에 의해 25개의 마찰재가 제작되었으며, 축소 마찰 시험기로 마찰, 마모특성을 조사하였다. 아라미드 섬유, 흑연, 구리섬유는 열화에 대한 저항성을 향상시켰으며, 반면 삼황화 안티몬, 지르콘은 열화현상을 가속시키는 것으로 나타났다. 이러한 결과는 각 원료들이 계면에 미치는 영향이 각기 다르기 때문으로 사료된다. 아라미드 섬유, 흑연, 구리섬유등은 계면을 안정화 시켰으며, 삼황화 안티몬, 지르콘등은 고온에서 불안정한 계면을 형성시키는 것으로 나타났다.

Key words – constrained mixture design, fade, friction materials

1. Introduction

Friction materials in an automotive brake system should maintain effective and safe performance at a wide range of pressure, speed, temperature, humidity, and others. The performance of the friction material is not depended on a single ingredient, but determined by the integration of tribological effects from numerous constituents in the composite. Normally, a friction material consists of more than 10 ingredients such as binder resin, reinforcing fibers, solid lubricants, abrasives, fillers, and other friction modifiers [1] and types and relative amounts of the ingredients have been determined by empirical experiences.

The effect of each ingredient for the brake performance of the friction material has been intensively studied since the friction material is considered as one of the main causes of brake induced problems such as fade, judder, and noise. Among those brake related issues, the fade behavior at high temperatures attracted much attentions since it is closely related to the safety issues during severe brake applications. This is because the coefficient of friction is decreased as the temperature at the sliding interface is increased, so that a vehicle speed can't be controlled at high temperature. The fade phenomenon is often experienced by a driver when a vehicle descends from the top of hill while engaging a brake to maintain a vehicle speed.

The cause of the high temperature fade has been attributed to the thermal decomposition of organic ingredients and subsequent vaporization of the organics, which changes the contact area at the sliding interface [2,3]. However, the fade at high temperatures appears not solely dependent on the decomposition of

---

†주저자 · 책임저자 : hojang@korea.ac.kr

Table 1. Basic formulation of the friction material used in this work

Classification	Raw material	Content (vol.%)
Group 1	Phenolic resin Cashew Aramid pulp	44
Group 2	Graphite Sb_2S_3 MoS_2	10
Group 3	Potassium titanate $ZrSiO_4$ Copper fiber	26
Filler	$BaSO_4$	20
Sum		100

organic ingredients but also strongly affected by the properties other ingredients. This is because the physical properties and morphology of the ingredients bound by binder resin change the tribological properties at elevated temperatures. In this study, the constrained mixture design was used to find the factors that affect high temperature fade.

2. Experiments

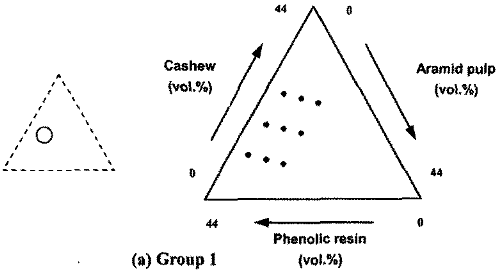
2-1. Composition and fabrication

Friction materials investigated in this study were low steel type materials containing 10 different ingredients. The composition of the friction material studied in this work is shown in Table 1.

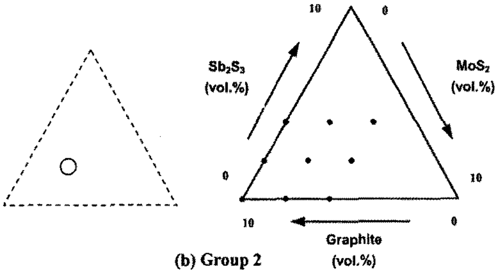
Friction material specimens were produced by a conventional procedure for a low steel friction material: dry-mixing, pre-forming, hot pressing, post-curing. The ingredients were divided into three groups in terms of expecting roles in a brake friction material (Table 1) and 25 friction materials with different formulations were prepared using the constrained mixture design [4,5,6] The polynomial regression was used to analyze the results. The data points from constrained mixture design were shown in Fig. 1 using ternary diagrams.

2-2. Physical properties

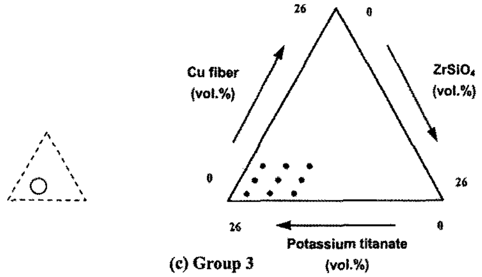
Hardness and porosity were measured to investigate the relation between the material property delivered by manufacturing conditions (temperature, pressure, time) of friction materials and friction property. The manufacturing procedure for the friction material specimen is shown in Table 2.



(a) Group 1



(b) Group 2



(c) Group 3

Fig. 1. Data points in the constrained mixture design from the material groups in the Table 1. (The center point indicates the base formulation.)

Hardness of friction material specimens was measured using a Rockwell hardness tester (Akashi, ARK600) in S scale. Porosity was obtained by a mercury porosimeter (Auto Pore II 9220) up to 30,000 psi. Thermal analysis (TGA/DTA) was performed to investigate the thermal decomposition of ingredients using a thermal analyzer (Netzsch model STA409EA) at 10°C/min up to 1000°C [7].

2-3. Tribotests

The friction coefficient and the specific wear rate

Table 2. Molding conditions used in this work

Specimen number	Hot pressing condition		
	Time (min.)	Temperature (°C)	Pressure (kgf/cm²)
1	9	180	270
2			295
3			320
4			345
5		200	270
6			295
7			320
8			345
9		220	270
10			295
11			320
12			345

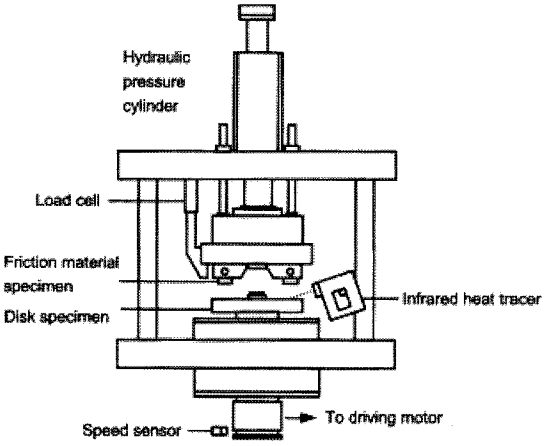


Fig. 2. A schematic of the pad-on-disc type tribotester.

were obtained with a small scale friction tester. A schematic of Krauss type friction test used in this study is shown in Fig. 2.

Friction force, applied pressure, disc temperature, and rotation speed were recorded in situ by a PC-based data acquisition system (LAB-PC-1200, National Instrument) during friction test The data acquisition rate was set at 100 Hz. Two pieces of friction materials which were cut in 2 cm × 2 cm × 1 cm were pressed against a disc using a hydraulic press. The counter disc used in the friction test was gray cast iron. The total apparent contact area on the disc surface was 8 cm^2. The temperature of the disc was measured using a non-contacting infrared thermometer (3M Scotchtrak™ IR-16). The detailed test procedure used for this study was listed in Table 3.

Table 3. Friction test procedures

Burnish	IBT: 100°C, 0.7 MPa, 150 sec, 5 times
Drag	IBT: 100°C, 0.7 MPa, 150 sec, 5 times
Constant Interval test	IBT: 100°C, 4 m/s, 0.7 MPa, 20 sec (drag) & 10 sec (off-drag), 20 times
Wear test	IBT: 100°C, 4 m/s, 0.7 MPa, 180 sec, 20 times

3. Results

3-1. Effect of molding conditions on physical properties

Hardness and porosity of a brake friction material are important physical properties that affect brake performance such as pedal feel during brake application and damping capacity. As a preliminary examination, those two important material properties were investigated as a function of temperature and pressures during molding. This is because the molding condition can change the mechanical property of the friction material by changing the molecular interaction of a binder resin [6].

Twelve specimens were produced by changing the molding temperature and applied pressure (Table 2). Fig. 3 shows the hardness and porosity plotted as functions of temperature and pressure. The figure shows that the hardness increases rapidly by increasing the pressure and temperature slightly increases the hardness, suggesting that applied pressure is more sensitive than the temperature to control the hardness of the friction material.

On the other hand, the porosity did not show significant changes with molding temperature and pressure. This indicates that binding capability of resin is enhanced more effectively by increasing pressure since real contact areas between binder resin and other ingredients are increased at high pressure molding condition.

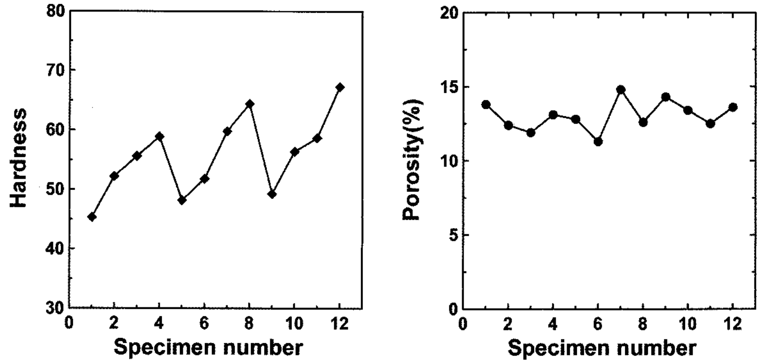


Fig. 3. (a) Hardness and (b) porosity of friction materials with specimen numbers.

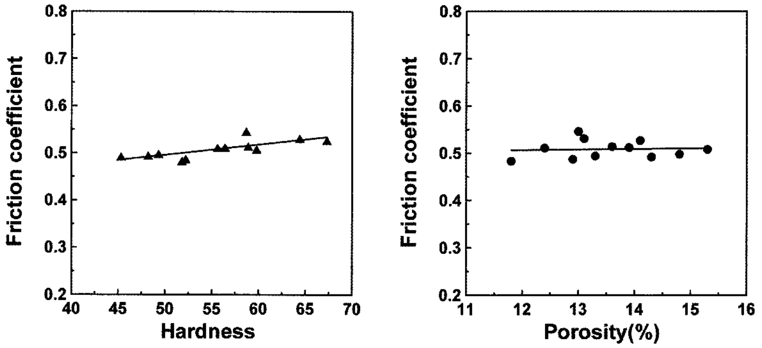


Fig. 4. Correlation of the friction coefficient with (a) hardness and (b) porosity.

The effects of hardness and porosity on the coefficient of friction were also examined (Fig. 4). The figure shows that the friction coefficient tends to increase with hardness but the coefficient of friction is independent with porosity. The slight influence of hardness on the coefficient of friction seems related to the binding force of the abrasives with binder resin.

3-1. Fade resistance

Fade resistance of the friction materials were measured by calculating the difference between maximum and minimum coefficients of friction obtained during the constant interval tests (CIT) as described in Table 3. The friction materials produced by the constrained mixture design were tested in 3 groups. The group 1 was consisted of nine friction material specimens with different compositions as functions of the three organic ingredients. The specimens in the group 2 were composed of nine specimens containing different relative amounts of solid lubricants and by the different relative amounts of other three friction modifiers group 3 was designed.

3-1-1. Group 1 (Organics)

According to the constrained mixture design, the composition of phenolic resin, cashew, and aramid pulp were changed by ±50% and produced nine different specimens by fixing other ingredients. After a series of CIT examination, the amounts of fade ($\Delta\mu = \mu_{max} - \mu_{min}$) obtained from the nine specimens were plotted in the Fig. 5 after polynomial regression (coefficient of determination: $R^2 = 0.91$). The figure shows that aramid pulp is more resistant to heat during high temperature drag test than binder resin and cashew particles used in the brake friction material.

3-2-2. Group 2 (solid lubricants)

The same type of a mixture design was repeated

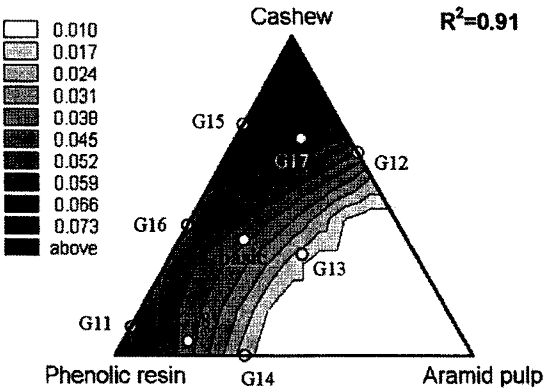


Fig. 5. $\Delta\mu$ $(=\mu_{max}-\mu_{min})$ **on a ternary diagrams enclosed by the confined compositions among group 1.**

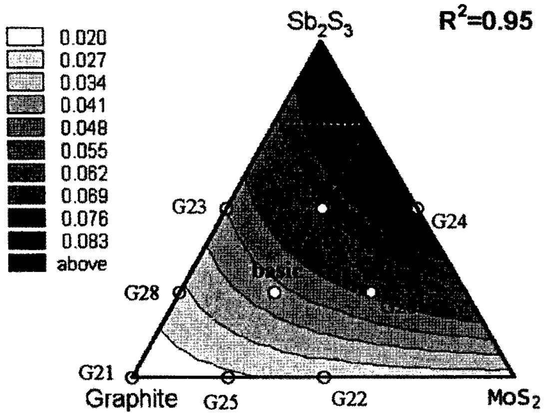


Fig. 6. $\Delta\mu$ $(=\mu_{max}-\mu_{min})$ **on a ternary diagrams enclosed by the confined compositions among group 2.**

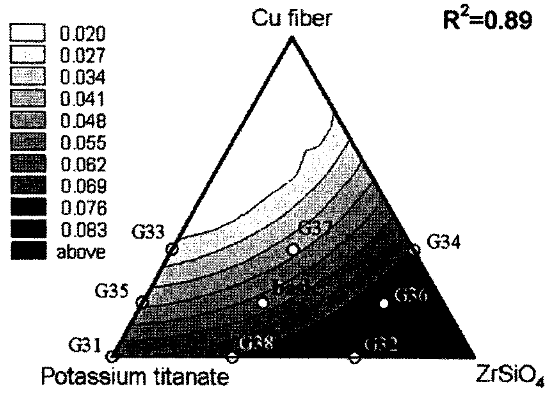


Fig. 7. $\Delta\mu$ $(=\mu_{max}-\mu_{min})$ **on a ternary diagrams enclosed by the confined compositions among group 3.**

using solid lubricants; graphite, Sb_2S_3, and MoS_2. Fig. 6 shows the results from constant interval tests after polynomial regression (coefficient of determination: $R^2=0.95$). The figure suggests that graphite is more resistant to fade than two others and molybdenum disulfide has better heat resistance than antimony sulfide

3-2-3. Group 3

Fade resistance of potassium titanate, $ZrSiO_4$, and Cu fiber were also examined by changing their compositions by ±50%. Fig. 7 shows the result of the polynomial regression and the coefficient of determination ($R^2=0.89$). It indicates that Cu fiber in the friction material improves fade resistance than other two inorganic ingredients.

3-3. Wear test

Wear test was performed to measure wear amount as a function of the composition. The specific wear rate was obtained by normalizing the wear amount by energy (friction force × sliding distance) associated during the wear test. The specific wear rates of all the specimens were shown in Fig. 8.

The figure suggests that the specimens G12, G15, and G17 in the group 1, which contain larger amounts of cashew than the others show bigger wear rate. In the group 2, G21, G25, and G28 that contain more graphite exhibite lower wear rates. In the group 3, G32, G34, and G36 with higher contents of $ZrSiO_4$ are observed to have bigger wear rate.

4. Discussion

The drag tests in constant interval mode and the wear test mode were performed to examine the influence of the ingredients at high temperature fade mode. The result shows that aramid pulps help to improve the fade resistance more than other organic ingredient. This suggests that the fibril aramid pulp increases the reinforcement of the other ingredients and slow down thermal disintegration of the friction materials. A similar effect from aramid pulp in the brake friction material was reported in the previous publication [3]. It also

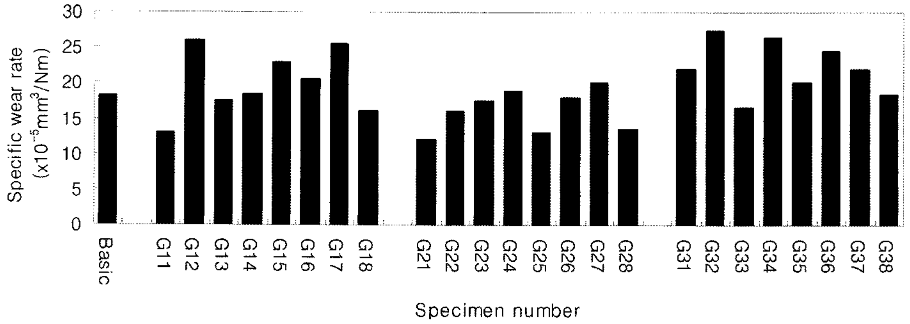
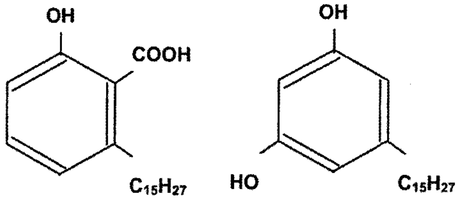


Fig. 8. Specific wear rates of friction materials normalized by friction energy.

showed the improvement of fade resistance by increasing the aramid pulp and synergistic effects with whisker fibers.

The fade resistance of the friction material was worsened by increasing the amount of cashew particles. The deterioration of heat resistance by cashew is attributed to its low thermal decomposition temperature. Although the cashew particles are added to the friction material to improve the friction effectiveness at low temperatures and to improve the damping capacity, this result indicates too much content can diminish the fade resistance of the friction materials at severe braking conditions. To investigate thermal decomposition of cashew, thermogravimetry analysis (TGA) was carried out (Fig. 9). It shows that weight loss begins near 200°C and followed by dramatic thermal decomposition beyond 400°C. Cashew is produced from cashew nut shell liquid (CNSL) which is a natural product from cashew nut trees. CNSL is composed with 90% of anacardic acid and 10% of cardol and a very small amount of cardanol and methyl cardol (Fig. 10). The low temperature decomposition of cashew is attributed to the easy decay of these anacardic acid and cardol molecules.

The better fade resistance of graphite than other solid lubricant is because of its higher decomposition temperature. Fig. 11 shows the results from TGA,



(a) (b)

Fig. 10. Chemical structure of (a) anacardic acid and (b) cardol.

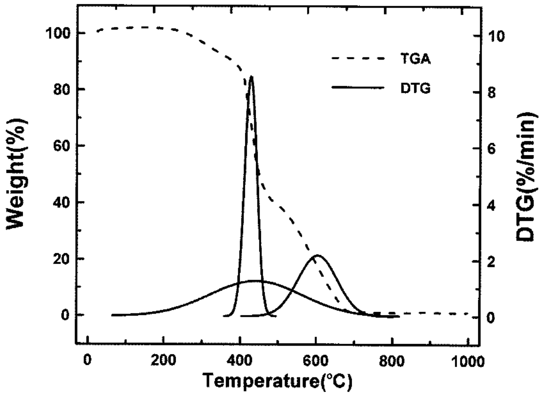


Fig. 9. Weight loss and derivative weight loss curves of cashew.

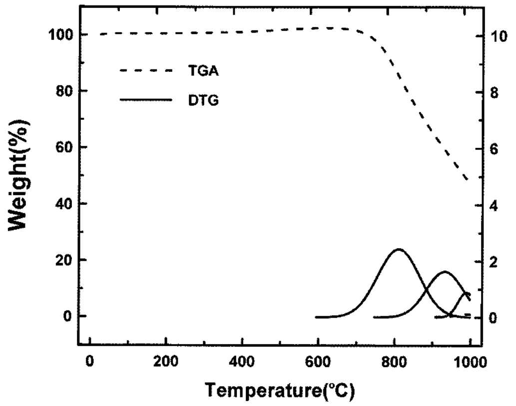


Fig. 11. Weight loss and derivative weight loss curves of graphite

indicating that the thermal decomposition begins near 700°C. On the other hand, the poor fade resistance of the MoS_2 and Sb_2S_3 is attributed to the oxides produced by oxidation at elevated temperatures and aggravation at the sliding interface [8,9]. The oxides present at the sliding interface after oxidation of the two chalcogenide compounds also worsened the wear rate since they played as abrasive particles.

Improvement of fade resistance by Cu fibers is attributed to its high thermal conductivity. Enhanced thermal diffusivity at the sliding surface appears to prevent the local heating of the contact plateaus and reduce the thermal distortion of the counter disks. However, Cu fibers do not reduce the wear rate since that are oxidized at high temperatures and can provide oxide particles, which are detrimental to wear resistance. However, the bad oxide effect appears to be compensated with another role of Cu since Cu helps to produce the transfer film on the gray iron brake disks.

5. Conclusions

Using the constrained mixture design, the relation between the high temperature fade resistance and the relative amounts of ingredients was investigated. Constant interval tests and wear tests were performed to find ingredients that strongly affect the fade amount ($\Delta\mu = \mu_{max} - \mu_{min}$) and wear rate. The results were as follow:

1. Hardness of the friction material was more sensitive to the applied pressure than temperature during molding. On the other hand, porosity was sensitive to the molding temperature and pressure. The coefficient of friction was slightly increased with hardness but porosity did not change the friction coefficient.

2. Among organic ingredients, cashew aggravated the high temperature fade behavior while aramid improves the fade resistance reduced.

3. Graphite, a solid lubricant, acted as an anti-fade material with good wear resistance and high thermal stability.

4. Cu fiber with high thermal conductivity showed good anti-fade property with the tendency of forming the transfer film.

Acknowledgements

This work was supported by the Korea Science and Engineering Foundation (KOSEF) through the National Research Lab. Program funded by the Ministry of Science and Technology (No. R0A-2007-000-10011-0)

References

1. Jacko MG and Rhee SK, “Brake Lining and Clutch Facings”, Kirk-Othmer encyclopedia of chemical technology, Vol. 4. 4th ed, 1992.
2. J.D. Halderman, “Automotive Brake Systems”, Prentice Hall, Columbus, Ohio, 1996.
3. Kim, S.J., Cho, M.H., Lim, D.S., and Jang, H., “Synergistic Effects of Aramid Pulp and Potassium Titanate Whiskers in the Automotive Friction Material”, *Wear*, Vol. 251, pp. 1484-1491, 2001.
4. Jang, H., Lee, J.S., and Fash, J.W., “Compositional Effects of the Brake Friction Material on Creep Groan Phenomena,” *Wear*, Vol. 251, pp. 1477-1483, 2001.
5. J.A. Cornell, “Experimental Design with Mixture: Design, Models and the Analysis of Mixture Data”, 2nd Edition, Wiley, New York, 1990.
6. D.C. Montgomery, “Design and Analysis of Experiments,” 3rd Edition, Wiley, New York, 1991.
7. Kim, S.J. and Jang, H., “Friction and Wear of Friction Materials containing Two Different Phenolic Resins Reinforced with Aramid Pulp”, *Tribology International*, Vol. 33, No. 7, pp. 477-484, 2000.
8. Kim, S.J. and Jang, H., “Effects of the Relative Amounts of Graphite and Antimony Trisulfide (Sb_2S_3) on Brake Performance of Non-Asbestos Organic(NAO) Type Brake Linings”, *Journal of the KSTLE*, Vol. 17, No. 5, pp. 351-357, 2001.
9. Kim, S.J. and Jang, H., “The Effects of Antimony Trisulfide (Sb_2S_3) and Zirconium Silicate ($ZrSiO_4$) in the Automotive Friction Material on Friction Characteristics”, *Wear*, Vol. 239, No. 2, pp. 229-236, 2000.
10. H. Jang and S.J. Kim, “Brake Friction Materials”, in Ch.14 of “Polymer Tribology”, edtied by S.K. Shinha and B.J. Briscoe, Imperial College Press, London, 2009.