

Research Article

Shape of Field-Induced Nanostructures Formed by STM

Subhashis Gangopadhyay, Asit Kumar Kar, and Balbir Kumar Mathur

Department of Physics and Meteorology, Indian Institute of Technology, Kharagpur 721302, India

Received 13 September 2006; Accepted 11 October 2006

Recommended by Rakesh K. Joshi

Creation of controlled and reproducible nanostructures on material surfaces using scanning tunneling microscope is a novel technique, which can be used for a variety of applications. We have examined the shape of the nanostructures so formed on the gold film using tungsten tip and examined the formation parameters, which govern their shape and size. During our investigations it is found that the reproducibility of mound formation can reach up to 90% under optimum operating conditions, whereas the pit formation can be made with almost 100% reproducibility. Formation mechanism of such nanostructures is also discussed.

Copyright © 2007 Subhashis Gangopadhyay et al. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

1. INTRODUCTION

Formation of field-induced nanostructures on thin films and on plane surfaces has been carried out by a large number of groups [1–5]. Controlled and reproducible modification of surfaces on a nanometer scale or even on an atomic scale has been projected to be of great technological as well as fundamental importance. Theoretical investigations on the mechanism of nanostructure formation are quite informative and provide substantial insight.

Different techniques to modify the surface using STM can broadly be classified into three categories. In the first category, atoms are transferred at specific sites using electrostatic adherence of atoms to the tip [6, 7]. Second technique is to create mounds and pits on the substrate by locally destroying the surface, that is, mechanically scratching the surface with the tip [8]. And the third is to produce the same structure by applying a voltage pulse between the tip and the sample surface [9–23]. In the process of mechanical scratching, the tip loses its sharpness, thereby making it ineffective for subsequent topographic application. But in the tip-bias pulsing method, it is well controlled and nondestructive. In this pulse-writing method, the main control parameters are the sign, magnitude, duration, and the number of voltage pulse applied between the tip and sample surface.

The most used technique is the bias-pulse technique. Nanostructures formed by this technique can again be classified into two broad categories: the pit (valley) [19, 23] and the mound (hill) [7, 10, 12–15, 17, 18]. In case of forming

a pit, the sample material is scooped out from the surface. There are various processes to take out the sample from the surface such as mechanical contact [10, 13] or evaporation of the material by field effect or local melting [9, 11, 12]. In the case of mound formation, the sample is added to the surface [15] or some plastic deformation [14] may take place.

Formation of a pit or a mound is reproducible to a very large extent as has been reported in a large number of publications and during our own investigations. But what concerns us at this stage is that at times we attempt to form a pit but gets a mound instead or sometimes the other way around. With known parameters controlling the metamorphism (like pulse height, polarity, duration, repetition, etc.), we have attempted to create many nanostructures on gold films by bias-pulse technique using a tungsten tip. We describe the most possible formation of pit and mound and its dependence on parameters like the pulse height, polarity, set current for the pulse, and the nature of the tip structure. STM is operated in air ambient at room temperature and the gold film is chosen because of its lower threshold for field evaporation and oxide protective inert nature. We also discuss the possible physical mechanism regarding the phenomenon.

2. EXPERIMENT

Thin films of gold (500 Å) have been deposited on the various substrates (silicon, mica, and glass), by thermal evaporation method in the physical vapor deposition (PVD) unit, at a base pressure of 10^{-6} T at room temperature. The

films are subsequently transferred into the STM (model UHV- 635, RHK Technology, Inc.) chamber. An electrochemically etched [24] tungsten wire of diameter 0.35 μm is used as a STM tip for the topographic tunneling and nano-modification of the gold surface. Applying bias pulse between the tip and the surface nanoscale structures has been created. For the topographic data recording, the STM is operated in the constant current mode using a feed back loop. Throughout the scanning, the bias voltage is maintained at a fixed value. The tip is scanned over the surface and the topographic data is stored as 256×256 data point in the SM2 file format.

The movement of the tip and the sample are well controlled by a piezo tube. The sample, fixed on sample holder, is placed on a piezo tube and the position of the sample below the tip can be adjusted by controlling the voltage applied to the offset piezo. This facility permits us to shift the sample in a precisely controlled manner so as to position the tip at a required location at the surfaces.

In our STM, the tip is grounded and the bias voltage is supplied to the sample surface. The STM is operated at various bias voltages (~ 100 mV) and also the tunneling current (~ 100 pA) is varied to get the good topographic image. Prior to the surface modification, smooth areas are selected by scanning the large areas at different locations on the sample surface. To form the nanostructure the bias pulse is applied in the range of 2–10 V and the duration of each pulse is kept fixed at 1 ms. To study the dependence of the shape of the nanostructure on tip-sample separation, the set current is varied for various pulses. The probability of formation of mound and pit is dependent on the height of the applied pulse. A statistical analysis of the probability of formation of mound and pit is done applying the pulses of various heights. The spectroscopic studies of the surface materials are done before and after formation of mound by applying a bias pulse.

3. RESULTS AND DISCUSSION

Nanostructures created by bias-pulse technique ranges from simple mound and pit to complex formation. For certain settings, the probability of reproducibility goes as high as 90% whereas for others it may be lower. Figure 1 illustrates the point that using the same tip, under similar operating conditions different surface modifications can be formed. Figures 1(a) and 1(b) are clear cases of mound formation but Figure 1(c) depicts a damage grain boundaries. At another location four pulses were applied and grain coalescence appears to have taken place (Figure 1(d)). In only 10% cases unclear pit formation was detected (Figure 1(e)). As the pulse height is increased to 4 V, the probability of clear mound formation increases to 90%. At 5 V, the probability of mound formation decreases and pits become clearer (Figure 2). Figure 2(a) is a clear case of mound formation whereas Figures 2(b) and 2(c) are complex formations. The pit seen in Figure 2(d) is also formed while operating the system under similar conditions as in Figures 2(a), 2(b), and 2(c). As the bias pulse increases, the probability of pit formation increases. Figure 3 shows the probability of a different

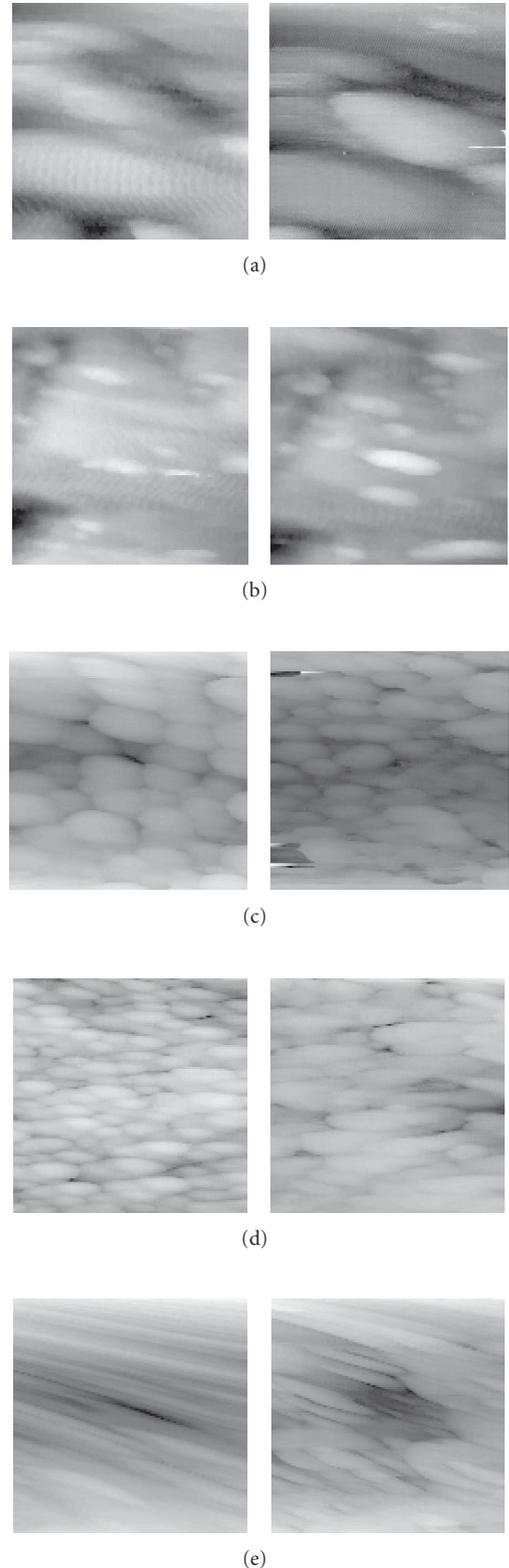


FIGURE 1: Surface topographs of the gold films on silicon, before (left side) and after (right side) applying the bias pulse of 3 V with tip-surface separation corresponding to tunneling current of 300 pA. Scan areas are (a) 100×100 nm²; (b) 200×200 nm²; (c) 250×250 nm²; (d) 300×300 nm²; and (e) 500×500 nm², respectively.

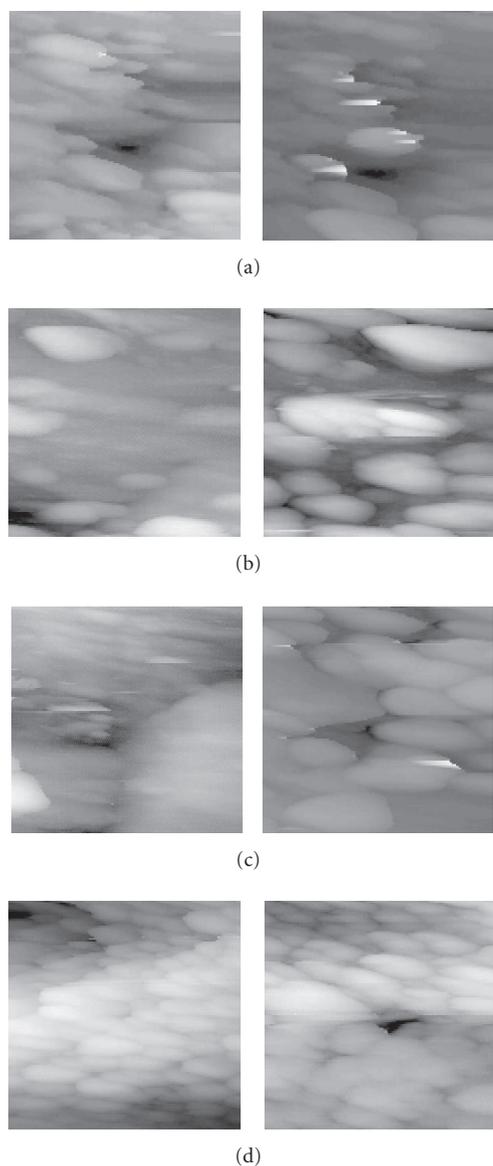


FIGURE 2: Surface topographs of the gold films on silicon, before (left side) and after (right side) applying the bias pulse of 5 V with tip-surface separation corresponding to tunneling current of 300 pA. Scan areas are (a) $200 \times 200 \text{ nm}^2$; (b) $200 \times 200 \text{ nm}^2$; (c) $200 \times 200 \text{ nm}^2$; and (d) $300 \times 300 \text{ nm}^2$, respectively.

distinct nanostructure formation, which could occur due to bias pulse.

This raises the question whether any other considerations determine the reproducibility. In this simple case study, gold films had been deposited on pieces of silicon wafer cut out from the same 4-inch wafer obtained from m/s Wafer World Inc., USA, under identical operating conditions. Surface topographs taken before the application of bias pulse were also identical. One way to generalize it is to consider different types of surface modifications under the same mechanism. It may be local melting, local plastic deformation due to sudden change of electrostatic field, or may be local transfer of

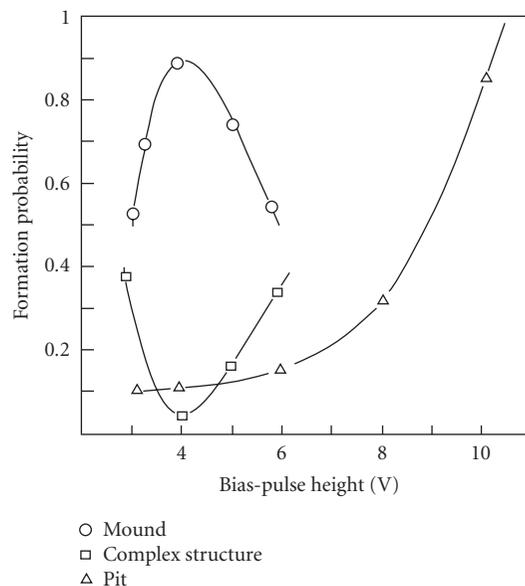


FIGURE 3: Dependence of the formation probability of different type of nanostructures on bias pulse.

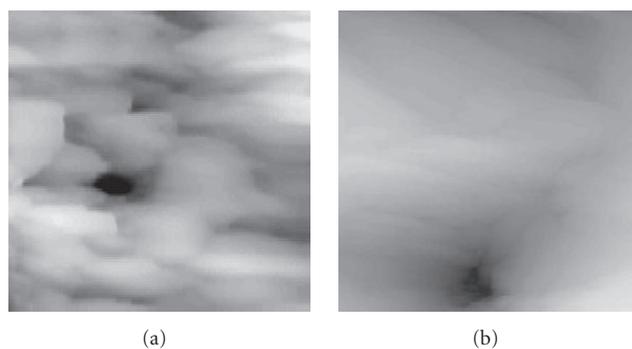


FIGURE 4: Change in the pit shape due to change in tip-surface distance. Both are taken using 3 V pulse but in (a) the tip-surface separation corresponds to tunneling current of 300 pA (scan area $280 \times 280 \text{ nm}^2$) while in (b) the tip-surface separation corresponds to 2 nA tunneling current (scan area $500 \times 500 \text{ nm}^2$).

material. Hence, the shape of the nanostructures so formed must depend on local defect structure of the film.

Another important consideration is the tip-surface separation. If this separation is decreased (by increasing set-tunneling current) the formations are well defined. All modifications shown in Figures 1 and 2 were carried out at setting the tip-sample separation corresponding to tunneling current of 0.3 nA. Now we compare pits formed at set currents of 0.3 nA and those formed at 2.0 nA as shown in Figure 4. The crater in Figure 4(b) is deeper (33 nm) and wider (100 nm) as compared to the one in Figure 4(a).

In order to investigate the effect of substrate, we have carried out similar experiment of films deposited on mica and

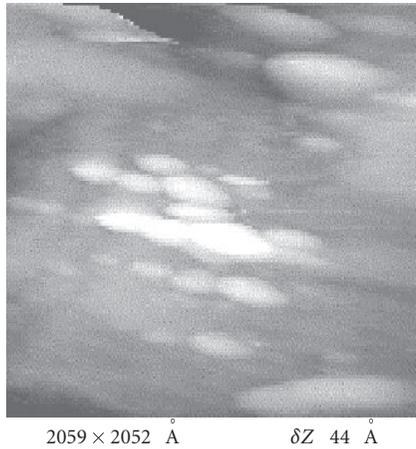
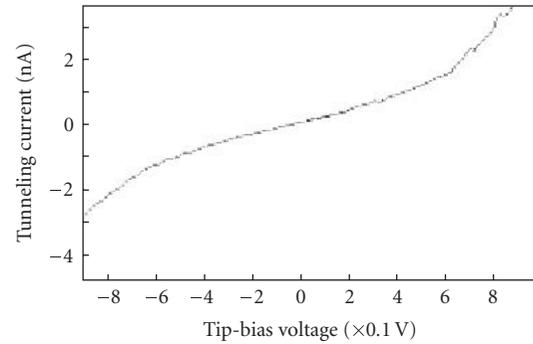


FIGURE 5: Multiple mound formation (scan area $200 \times 200 \text{ nm}^2$).

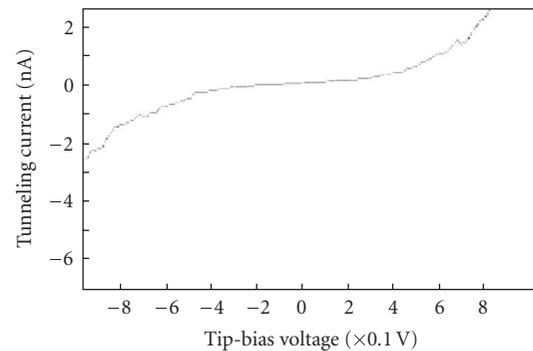
glass substrates. Results are similar and do not justify inclusion of additional topographs.

We illustrate two main factors, which determine the formation of good nanostructures. Looking at Figure 5, it is seen that a multimound formation is repeated at many locations of the topograph. These are produced due to application of single bias pulse. The possible causes of this defect are (a) the tip is likely to have three sharp-pointed corners and (b) some sort of switch debounce must have taken place in the electronics and an associated shake in either the tip-position or the surface or both. The multipoint tip does not effect the topography because the tunneling takes place predominantly from the point closest to the surface. But at the time of high field pulse applied between the tip and the sample, all the pointed corners generate different fields on the surface, thereby, creating multiple hillock of different size [14].

Our investigations support the theory proposed by Tsong regarding mechanism of nanostructure formation [9, 11, 12]. Such metamorphism is attributed to a local melting of the sample surface. It is seen that for the lower value of voltage pulse the probability of formation of mound is dominating. That can be explained as due to the voltage pulse, a high electric field is generated causing the local melting of the gold surface. Due to the high electrostatic force, the molten material is attracted towards the tip and creates a plastic deformation instead of pure elastic deformation [14]. In case of higher voltage pulse it is seen that the probability of pit formation is dominating. In this case the electrostatic field is sufficiently high to cause local field evaporation instead of melting. Formation of mound is not likely to be due to the material transfer from tip to surface, which can be explained as follows. We observed that even after repeated use of tip in the field application mode, the tip quality does not deteriorate and is capable of producing good quality reproducible topographs [14, 16–18]. Secondly, the possibility of transfer of atoms by field evaporation between tip and sample atoms is depending on the binding energy. As the binding energy of tungsten atom is greater than that of gold [9], the transfer of tip material (tungsten) to the surface (gold) is least likely. An-



(a)



(b)

FIGURE 6: Tunneling spectra before and after formation of a mound.

other support, which negates material transfer, is the similarity between tunneling I-V spectra obtained before and after the formation of nanostructure and with tip positioned on the mound (Figure 6). Slight change in the STS spectra is likely to be caused by the change in electronic state of the surface atoms due to high voltage pulse.

4. CONCLUSION

Using scanning tunneling microscope, different types of nanostructures have been formed. The possibility of the formation of various structures (mound, pit, and complex structures) with different pulse height has been studied. It is seen that for gold film on silicon, the probability of mound formation is higher for bias-pulse height below 6 V. At higher voltage, a pit is more likely to form. The effect of set current on nanostructure formation is also investigated. The possible mechanism behind the nanostructure formation is attributed to local melting (mound formation) for lower bias-pulse voltage and to local vaporization (pit formation) for higher bias-pulse voltage.

ACKNOWLEDGMENT

We are grateful to the Department of Science and Technology, Council of Scientific and Industrial Research, Government of India, for financial assistance in this project.

REFERENCES

- [1] S. P. Wilks, T. G. G. Maffei, G. T. Owen, K. S. Teng, M. W. Penny, and H. Ferkel, "Charge writing on the nanoscale: from nanopatterning to molecular docking," *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures*, vol. 22, no. 4, pp. 1995–1999, 2004.
- [2] T. G. G. Maffei, G. T. Owen, M. W. Penny, H. S. Ferkel, and S. P. Wilks, "STM patterning of SnO₂ nanocrystalline surfaces," *Applied Surface Science*, vol. 234, no. 1–4, pp. 2–10, 2004.
- [3] K. Sattler, "Nanolithography using the scanning tunneling microscope," *Japanese Journal of Applied Physics*, vol. 42, no. 7 B, pp. 4825–4829, 2003.
- [4] Th. Schimmel, H. Fuchs, S. Akari, and K. Dransfeld, "Nanometer-size surface modifications with preserved atomic order generated by voltage pulsing," *Applied Physics Letters*, vol. 58, no. 10, pp. 1039–1041, 1991.
- [5] E. S. Snow, P. M. Campbell, and P. J. McMarr, "Fabrication of silicon nanostructures with a scanning tunneling microscope," *Applied Physics Letters*, vol. 63, no. 6, pp. 749–751, 1993.
- [6] D. M. Eigler, C. P. Lutz, and W. E. Rudge, "An atomic switch realized with the scanning tunnelling microscope," *Nature*, vol. 352, no. 6336, pp. 600–603, 1991.
- [7] D. M. Eigler and E. K. Schweizer, "Positioning single atoms with a scanning tunnelling microscope," *Nature*, vol. 344, no. 6266, pp. 524–526, 1990.
- [8] U. Landman and W. D. Luedtke, "Consequences of tip-substrate interactions," in *Scanning Microscopy III*, R. Wiesendanger and H. J. Guntherodt, Eds., chapter 9, p. 207, Springer, Berlin, Germany, 1993.
- [9] T. T. Tsong, "Effects of an electric field in atomic manipulations," *Physical Review B*, vol. 44, no. 24, pp. 13703–13710, 1991.
- [10] J. I. Pascual, J. Méndez, J. Gómez-Herrero, A. M. Baró, N. García, and V. T. Binh, "Quantum contact in gold nanostructures by scanning tunneling microscopy," *Physical Review Letters*, vol. 71, no. 12, pp. 1852–1855, 1993.
- [11] U. Gratzke and G. Simon, "Mechanism of nanostructure formation with the scanning tunneling microscope," *Physical Review B*, vol. 52, no. 11, pp. 8535–8540, 1995.
- [12] U. Gratzke and G. Simon, "New mechanism of nanostructure formation with the STM," *Microelectronic Engineering*, vol. 27, no. 1–4, pp. 35–38, 1995.
- [13] R. Taylor, R. S. Williams, V. L. Chi, et al., "Nanowelding: tip response during STM modification of Au surfaces," *Surface Science*, vol. 306, no. 1–2, pp. L534–L538, 1994.
- [14] V. Srinivas, M. V. H. Rao, B. K. Mathur, and K. L. Chopra, "Creation of nanostructures on nickel thin films by STM," *Bulletin of Materials Science*, vol. 17, no. 6, pp. 841–848, 1994.
- [15] R. S. Becker, J. A. Golovchenko, and B. S. Swartzentruber, "Atomic-scale surface modifications using a tunnelling microscope," *Nature*, vol. 325, no. 6103, pp. 419–421, 1987.
- [16] H. J. Mamin, S. Chiang, H. Brik, P. H. Guethner, and D. Rugar, "Gold deposition from a scanning tunneling microscope tip," *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures*, vol. 9, no. 2, pp. 1398–1402, 1991.
- [17] H. J. Mamin, P. H. Guethner, and D. Rugar, "Atomic emission from a gold scanning-tunneling-microscope tip," *Physical Review Letters*, vol. 65, no. 19, pp. 2418–2421, 1990.
- [18] S. E. McBride and G. C. Wetsel Jr., "Nanometer-scale features produced by electric-field emission," *Applied Physics Letters*, vol. 59, no. 23, pp. 3056–3058, 1991.
- [19] Y. Z. Li, L. Vazquez, R. Piner, R. P. Andres, and R. Reifenberger, "Writing nanometer-scale symbols in gold using the scanning tunneling microscope," *Applied Physics Letters*, vol. 54, no. 15, pp. 1424–1426, 1989.
- [20] U. Staufer, R. Wiesendanger, L. Eng, et al., "Surface modification in the nanometer range by the scanning tunneling microscope," *Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films*, vol. 6, no. 2, pp. 537–539, 1988.
- [21] U. Staufer, R. Wiesendanger, L. Eng, et al., "Nanometer scale structure fabrication with the scanning tunneling microscope," *Applied Physics Letters*, vol. 51, no. 4, pp. 244–246, 1987.
- [22] J. Schneir, R. Sonnenfeld, O. Marti, P. K. Hansma, J. E. Demuth, and R. J. Hamers, "Tunneling microscopy, lithography, and surface diffusion on an easily prepared, atomically flat gold surface," *Journal of Applied Physics*, vol. 63, no. 3, pp. 717–721, 1988.
- [23] I.-W. Lyo and Ph. Avouris, "Field-induced nanometer- to atomic-scale manipulation of silicon surfaces with the STM," *Science*, vol. 253, no. 5016, pp. 173–176, 1991.
- [24] A. K. Kar, S. Gangopadhyay, and B. K. Mathur, "Reverse electrochemical floating-layer technique of SPM tip preparation," *Measurement Science and Technology*, vol. 11, no. 10, pp. 1426–1431, 2000.



Hindawi

Submit your manuscripts at
<http://www.hindawi.com>

